

Exhibit 8

Dr. William E. Longo

Dr. William E. Longo

VITAE

William Edward Longo, Ph.D
MAS, LLC
3945 Lakefield Court
Suwanee, Georgia 30024
Work Telephone: (770) 866-3200

EDUCATION

October 1980 to December 1983	Received Doctor of Philosophy in Materials Science and Engineering, University of Florida
June 1979 to October 1980	Completed requirements for Master of Science in Materials Science and Engineering, University of Florida
September 1972 to June 1977	Received Bachelor of Science degree; Major in Microbiology, Minor in Chemistry, University of Florida.

PROFESSIONAL WORK HISTORY

September 1987 to Present	President of MAS, LLC (previously Materials Analytical Services, Inc.) Suwanee, Georgia.
August 1987 to February 1988	President and Founder of Longo Microanalytical Services, Inc., Gainesville, Florida.
October 1983 to August 1987	President and Founder of Micro Analytical Laboratories, Inc., Gainesville, Florida.
March 1985 to December 1987	Visiting Assistant Professor; University of Florida, Department of Materials Science and Engineering.
August 1983 to March 1985	Post Doctoral Associate; University of Florida, Department of Materials Science and Engineering.



B.S. Microbiology
M.S. Materials Science and Engineering
Ph.D. Materials Science and Engineering

EPA Peer Review Group (Asbestos)
ASTM

Asbestos Testing Standards Authorship:
ASTM 5755

Laboratory Certified By:
NVLAP
AIHA
ISO
FDA Lab Number

Consulted for:
Port Authority (NY/NJ)
Various States and Municipalities
Corporations
CDC/NIH
NASA
Air Force

Samples Tested for Asbestos:
Approx. 400,000

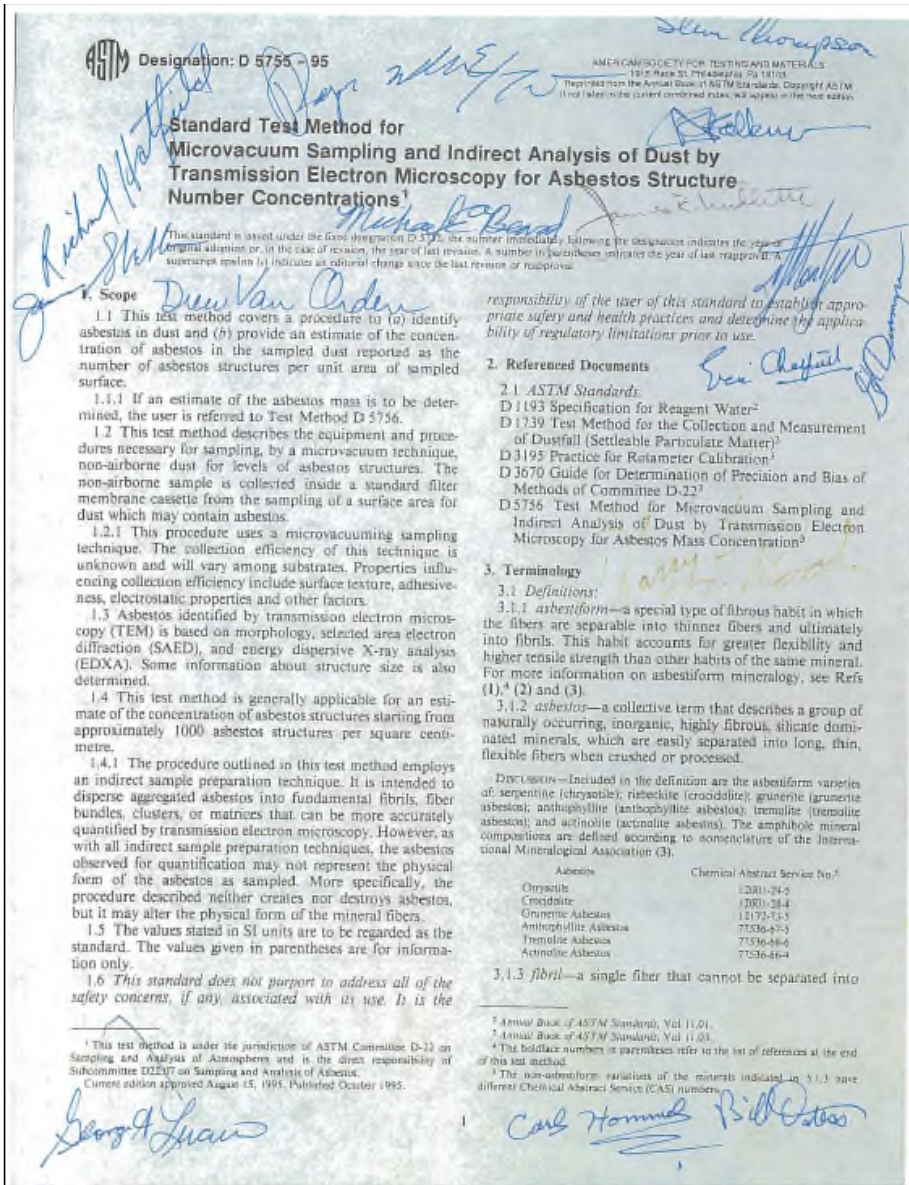


National Institutes
of Health

Tab 1, Dr. Longo CV

Dr. William E. Longo

- Leadership on ASTM D22 Committee and D22.07 Subcommittee on Sampling and Analysis of Asbestos
- Collaboration with EPA and Government and Industry Scientists
- Originally approved in 1995 based on MAS Lab Method



Peer-Reviewed Publications, Academic and Scientific Presentations

- Approx. 45 Peer-Reviewed Journal Articles & Presentations
- Majority on the subject of analysis for asbestos

William E. Longo, Ph.D
Page 2

PATENTS

U. S. Patent Serial No. 4671954 June 9, 1987. Goldberg, E.P., Iwata, H., and Longo, W.E., "Microspheres for Incorporation of Therapeutic Substances and Methods of Preparation Thereof."

U. S. Patent Serial No. 4871716, October 3, 1989. Goldberg, E.P., Longo, W.E., and McCluskey, R.A., "Magnetically Responsive, Hydrophilic Microspheres for Incorporation of Therapeutic Substances and Methods of Preparation Thereof."

PUBLICATIONS AND PRESENTATIONS

Egilman, D., Longo, W.E., "Egilman's Assessment Regarding Exposures of Auto Mechanics to "Amphiboles is Correct" *Inhalation Toxicology*, 2012: 24(9); 614-618.

Rigler, M.W., Longo, W.E. & Sauerhoff, M.W.: "Exposure to Fluoropolymers and VOCs during Spray Sealant Product Use" *Inhalation Toxicology*, 23 (11): 641-657, 2011.

Ewing, W.M., Hays, S.M., Hatfield, R., Longo, W.E. & Millette, J.R. "Zonolite Attic Insulation Exposure Studies" *Int. J. Occup. Environ. Health*, Vol. 16(3), Jul/Sep, 2010.

Rigler, M.W., Longo, W.E. Emission of Diacetyl (2,3 Butanedione) from Natural Butter, Microwave Popcorn Butter Flavor Powder, Paste, and Liquid Products", *Int. J. Occup. Environ. Health*, 16:291-302, 2010.

Rigler, M.W., Longo, W.E., "Qualitative Sulfur Gas Emission as a Specific Marker for Problematic Reactive Drywall, Proceedings of the Technical Symposium on Imported Corrosive Drywall, November 5-6, 2009, the University of Florida, Gainesville, FL.

Longo, W.E., Rigler, M.W., Russell, P.E., Vitarelli, J.P., Hoffman, E.M. & Johnson, H.M. Health Effects of Welding, "The Characterization of Welding Fume Particulates and Mn Bioavailability Studies for SMAW and FCAW Consumables" NIOSH, West Virginia, July 2005.

Harris M.D., Ewing, W.M., Longo, W.E., DePasquale, C., Mount, M.D., Hatfield, R.L. & Stapleton, R. "Manganese Exposure During Shielded Metal Arc Welding (SMAW) in an Enclosed Space" *J. Occup. & Environ. Hyg.* 2(8) 375-382, 2005.

Longo, W.E., Egeland, W.B., Hatfield, R.L., Stapleton, R., and Hubbard J., "Tremolite Analysis of Chrysotile Containing Friction and Gasket / Packing Products", ASTM Johnson Conference, Johnson Vermont, July, 2002.

Denial of Daubert/Frye Challenges to MAS

Recent Daubert, Kelly Frye, Robinson and Motions to Strike Denied in Court Concerning MAS's Work Practice Studies and the Testimony of Dr. William E. Longo, Richard L. Hatfield & Michael D. Mount, CIH, William B. Egeland, M.S., P.G., Mark W. Rigler, Ph.D.

Cause No.	Case Name	Location	Law Firm	Expert	
1)	92-11238-G	Darrell Wayne Caves v Keene Corp.	134 th Judicial District Dallas District Court	Baron & Budd	WEL
2)	45664-A	Wesley Roberts v Owens Corning Fiberglass Corp.	18 th JDC, Parish of Iberville State of Louisiana	Baron & Budd	RLH
3)	DV98-03696	James Blackburn v Dresser Industries	116 th District Court Dallas County, Texas	Baron & Budd	WEL RLH
4)	DV98-03696	James Blackburn v. Dresser Industries	Tyler, Texas	Baron & Budd	WEL
5)		Robert Alton Adcock v. Owens Corning Fiberglass	153 District Court Tarrant County, Texas	Silber Peariman	
6)	95-1922	William Arthur Brown v Borg Warner	County Court at Law No. 2 El Paso County, Texas	Baron & Budd	WEL
7)	97-16440 04/13/99	Dennis C. Eisenreich and Victoria I. Eisenreich v. Durabla Manufacturing Company, et al.	Civil Division – Asbestos Allegheny y County, Pennsylvania Hon. Robert P. Horgos	Goldberg, Persky & White	RLH
8)	99-2681-3 04/10/01	Billy Ray Meadows v United States Gypsum	McLennan County Waco, Texas	Ness Motley et al	WEL
9)	00-01428	Clyde A. Black, Sr. v Garlock, National Service and A.P. Green	Glynn Superior Court Brunswick, Georgia	Lane & Gossett	
10)	590-228	Benjamin D. Jones v. CSX	Federal Court Brunswick, Georgia	Lane & Gossett	
11)	591-226	James B. Ostein v CXSW	Federal Court Brunswick, Georgia	Lane & Gossett	
12)	98-01249-1	Lee Bailey v U.S. Gypsum	162 nd Court Dallas, Texas	Baron & Budd	WEL
13)	59078	Bobby Jean Thorne,	196 th Judicial District	Hendler Law Firm	

Page 1 of 16

Updated: 7/2/2019

- Daubert/Frye challenges denied in at least 175 cases over past 25+ years including State and Federal Courts
- Testified about findings of asbestos in JBP in California, Kentucky, Missouri, New Jersey, New York, Oklahoma, & South Carolina (17 cases)

Tab 2, List of Daubert/Frye Denials

Dr. Longo – J&J Asbestos Talc Trials 2017-2019

1. *Herford v. J&J and Imerys* – Judge C. Edward Simpson, Pasadena, CA , 2017
2. *Lanzo v. J&J and Imerys* – Judge Ana Viscomi, Middlesex Co., NJ, 2018
3. *Anderson v. J&J* – Judge Gloria White-Brown, West Covina, CA, 2018
4. *Bostic v. J&J* – Judge Jean Toal, Darlington Co., SC (and retrial), 2018
5. *Brick v. J&J and Imerys* – Judge Stephen Moloney, Los Angeles, CA, 2018
6. *Ingham v. J&J* – Judge Rex Burleson, City of St. Louis, MO, 2018
7. *Weirick v. J&J and Imerys* – Judge Margaret Oldendorf, Pasadena, CA, 2018
8. *Henry v. J&J* – Judge Ana Viscomi, Middlesex Co., NJ, 2018
9. *Allen v. J&J* – Judge Timothy Canning, Humboldt County, CA, 2018
10. *Leavitt v. J&J* – Judge Brad Seligman, Alameda, CA, 2019
11. *Olson v. J&J* – Justice Lebovits, New York, NY, 2019
12. *Pipes v. J&J* – Judge Susan Stallings, Oklahoma City, OK, 2019
13. *Blinkinsop v. J&J* – Judge Peter Mirich, Long Beach, CA, 2019
14. *Johnson v. J&J* – Judge Jean Toal, Darlington Co., SC, 2019
15. *Rimondi v. J&J* – Judge Ana Viscomi, Middlesex Co., NJ, 2019
16. *Schmitz v. J&J* – Judge Frank Roesch, Alameda, CA, 2019
17. *Hayes v. J&J* – Judge Angela McCormick, Louisville, KY, 2019

Tab 2, List of Daubert/Frye Denials

What is asbestos?

“Asbestos Minerals”

Table 3. Chemical formulas for the **asbestos minerals**

Chrysotile	$\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$
Amphibole { Tremolite	$\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2 - \text{Ca}_2\text{Mg}_4\text{FeSi}_8\text{O}_{22}(\text{OH})_2$
Actinolite	$\text{Ca}_2\text{Mg}_4\text{FeSi}_8\text{O}_{22}(\text{OH})_2 - \text{Ca}_2\text{Fe}_4\text{MgSi}_8\text{O}_{22}(\text{OH})_2$
Ferroactinolite	$\text{Ca}_2\text{Fe}_4\text{MgSi}_8\text{O}_{22}(\text{OH})_2 - \text{Ca}_2\text{Fe}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$
Amphibole { Anthophyllite	$\text{Mg}_7\text{Si}_8\text{O}_{22}(\text{OH})_2 - \text{Mg}_4\text{Fe}_3\text{Si}_8\text{O}_{22}(\text{OH})_2$
Cummingtonite	$\text{Mg}_7\text{Si}_8\text{O}_{22}(\text{OH})_2 - \text{Mg}_{4.9}\text{Fe}_{2.1}\text{Si}_8\text{O}_{22}(\text{OH})_2$
Grunerite	$\text{Mg}_{4.9}\text{Fe}_{2.1}\text{Si}_8\text{O}_{22}(\text{OH})_2 - \text{Fe}_7\text{Si}_8\text{O}_{22}(\text{OH})_2$
Riebeckite	$\text{Na}_2\text{Fe}^{2+}\text{Fe}_2^{3+}\text{Mg}_2\text{Si}_8\text{O}_{22}(\text{OH})_2 - \text{Na}_2\text{Fe}_3^{2+}\text{Fe}_2^{3+}\text{Si}_8\text{O}_{22}(\text{OH})_2$

Tab 3, McCrone Particle Atlas, Pg. 6

Fibrous + Amphibole = Asbestos

Table 1, Asbestos terminology

<u>Asbestiform mineral</u>		<u>Corresponding nonasbestiform mineral</u>
Chrysotile		Lizardite and antigorite
Fibrous tremolite	} Amphibole	Nonfibrous tremolite
Fibrous actinolite		Nonfibrous actinolite
Fibrous anthophyllite		Nonfibrous anthophyllite
Amosite		Cummingtonite and grunerite
Crocidolite		Riebeckite

Tab 3, McCrone Particle Atlas, Pg. 4

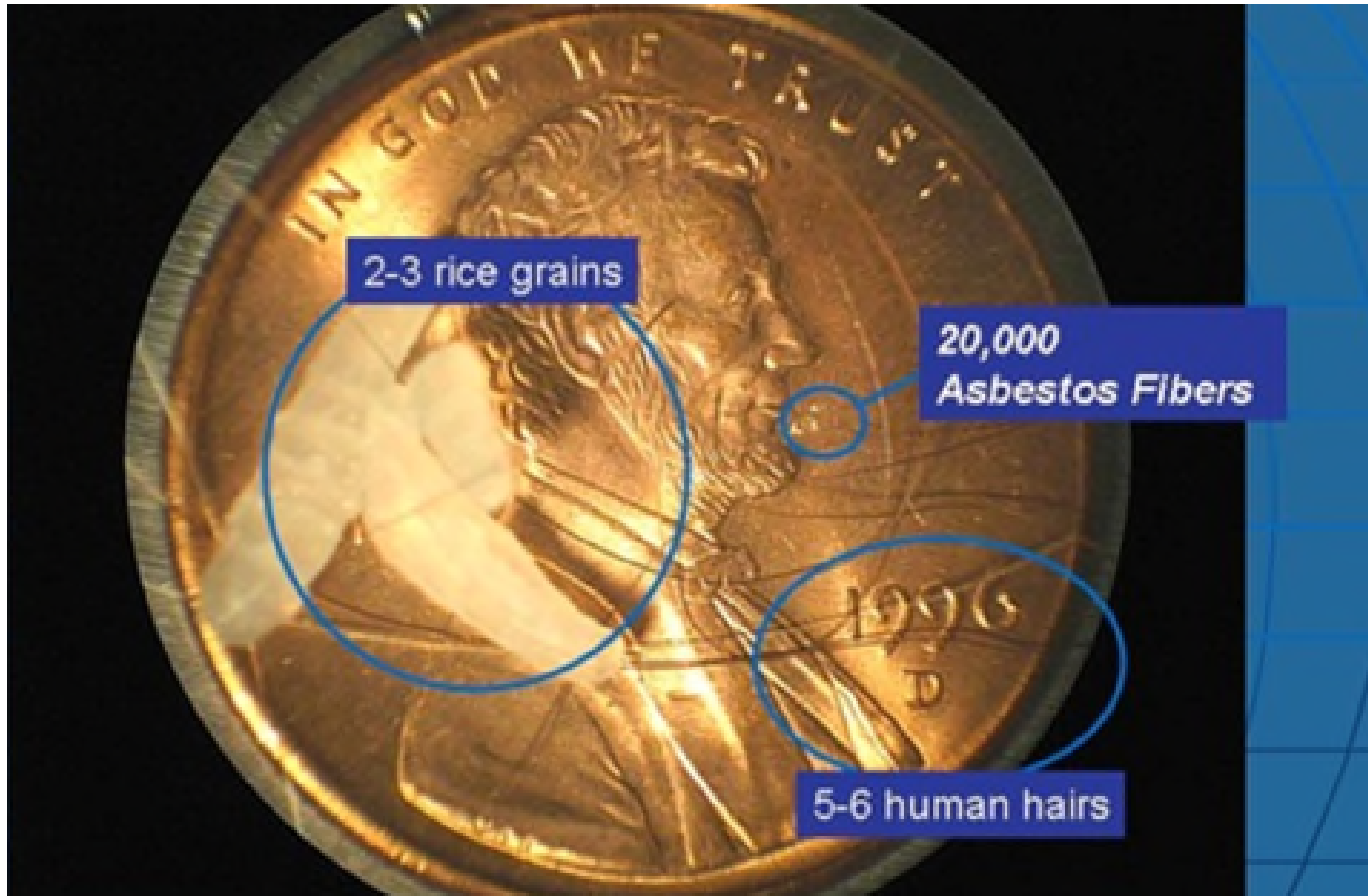
Fibrous = Fibers

i. *Fiber*. A structure having a minimum length greater than or equal to $0.5\ \mu\text{m}$ and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

$\geq 5:1$ Aspect Ratio
 $\geq 0.5\ \mu$ in length



Asbestos Fibers are Microscopic



Why Does Talc Need to be Tested for Asbestos?

Environmental Health Perspectives
Vol. 9, pp. 129-132, 1974

Asbestos in Talc

by Arthur N. Rohl*

NOTICE

THIS MATERIAL IS SUBJECT TO THE UNITED STATES COPYRIGHT LAW (TITLE 17, U.S. CODE) FURTHER REPRODUCTION IN VIOLATION OF THAT LAW IS PROHIBITED.

Talc deposits include asbestos minerals such as chrysotile and amphiboles that may be carried over into consumer products. Optical microscopy and x-ray diffraction analyses may not reveal their presence. Examples are given of electron microscopy procedures that permit detection and measurement.

Since the mining of talc rock almost invariably includes the mining of asbestos as well, the asbestos contaminant may be carried over into the consumer product and thus introduce the risk of asbestos disease. This possibility

Tab 8, Rohl 1974 "Asbestos in Talc," Pg. 129

MAS Testing of Historical J&J Talc Products & Source Talcs



The 71 J&J and Imerys-supplied historical cosmetic talcum powder containers/samples analyzed for this report, were chosen from the 1960's, 1970's, 1980's, 1990's and early 2000's.

The 71 product sample set consisted of 56 JBP/STS containers, and 15 historical Imerys' samples that were described as "railroad car" samples. The source of the talcum powder for these historical JBP/STS and Imerys containers/samples came from both the Italian (1960's, JBP/STS) and Vermont (1960's, 1970's, 1980's, 1990's, early 2000) talc mines. Included in this report are seven Asian Historical JBP samples that MAS analyzed from possibly only from the 1980's. The source of the talc that J&J used for these historical Asian samples was from the Dongyang talc mine in Korea.

Methods Used for Testing J&J Talc for Asbestos

Analytical Techniques Used in Peer-Reviewed Asbestos Analyses

[CANCER RESEARCH 55, 2232-2235, June 1, 1995]

Advances in Brief

Crocidolite Asbestos Fibers in Smoke from Original Kent Cigarettes¹

William E. Longo, Mark W. Rigler,² and John Slade

Materials Analytical Services, Inc., Norcross, Georgia 30092 [W. E. L., M. W. R.], and Department of Medicine, University of Medicine and Dentistry of New Jersey and St. Peter's Medical Center, New Brunswick, New Jersey 08901 [J. S.]



Applied Occupational and Environmental Hygiene
Volume 17(1): 55-62, 2002
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1047-322X/02 \$12.00 + .00

Fiber Release During the Removal of Asbestos-Containing Gaskets: A Work Practice Simulation

William E. Longo, William B. Egeland, Richard L. Hatfield, and Larry R. Newton
Materials Analytical Services, Inc., Suwanee, Georgia

Zonolite Attic Insulation Exposure Studies

**WILLIAM M. EWING, STEVE M. HAYS, RICHARD HATFIELD, WILLIAM E. LONGO,
JAMES R. MILLETTE**

INT J OCCUP ENVIRON HEALTH 2010;16:279-290

Tabs 10, 11, 12

Key Concepts – Testing for Asbestos in Talc

1. Preparation Method
2. Analytical Tool / Analysis Method
(TEM, PLM)
3. Limit of Detection / Sensitivity

Preparation Method: Heavy Liquid Separation

Preparation Methodology

Amphibole Content of Cosmetic and Pharmaceutical Talcs

by A. M. Blount*

Pharmaceutical and cosmetic-grade talcs were examined for asbestosiform amphibole content using a new density-optical method. Talcs under the Food and Drug Administration are not regulated as to asbestos content; however, all talcs were well below the level mandated by the Occupational Safety and Health Administration for industrial talcs. Only one was found to contain an amphibole particle size distribution typical of asbestos.

Introduction

In 1973 the Food and Drug Administration (FDA) proposed a regulation on the permissible asbestos content of talc (1). This regulation proposed to limit the amount of amphibole minerals to less than 0.1% and chrysotile to less than 0.01%. However, the optical microscopy method proposed was so complicated, lengthy, and subject to error that the proposed method was never finalized. Since then no final ruling has been issued.

The Occupational Safety and Health Administration, on the other hand, has been more rigorous and has instituted regulations despite the lack of methods to carry out the required measurements. One regulation, instituted in 1986, defines amphibole minerals as asbestos if the length to width ratio is 3:1 or greater. Because many nonfibrous cleavage fragments of amphibole minerals have a 3:1 aspect or greater and because there is no good evidence for adverse effects of these particles, a stay has been in effect on this part of the regulation (2). The second applicable regulation is the Hazard Communication Regulation (3), which applies to all chemicals used in the workplace. Specifically, it requires labeling of substances containing $\geq 1\%$ of a chemical hazardous to health and $\geq 0.1\%$ of a carcinogenic chemical.

Unfortunately, asbestos and amphiboles cannot be measured using currently developed methods to the level of 0.1% in the presence of talc. Some investigators have suggested that tremolite can be measured to that level by X-ray diffraction. But others have shown that the peak intensities vary between nonfibrous and fibrous tremolite (4) so that the 0.1% level of detection and measurement is doubtful except in cases where the sample has been spiked so that the exact nature of the tremolite is known. For anthophyllite there is little argument about the fact that detection cannot be made to 0.1%. However, the main problem with using X-ray diffraction for detection of amphibole minerals is that it gives no information about the shape of the particles, and shape is important in view of the uncertainty in the outcome of the asbestos regulation pertaining to nonfibrous amphiboles.

*Geology Department, Rutgers University, Newark, NJ 07102.

Environmental Health Perspectives
Vol. 94, pp. 225-230, 1991

The talcs that are pharmaceutical grade fall under the domain of the FDA and are therefore nonregulated in regard to fibrous mineral content. In the course of developing a technique to facilitate quantification of amphiboles in talc (5), pharmaceutical and high-grade talcs were examined. They were found to have very low amphibole content and, because of this, were extensively used in examining the lower limit of detection of the new method. The purpose of this paper is to describe the results of analyses for content and shape of amphibole mineral fragments in cosmetic and pharmaceutical talc powders of the United States.

Methods

The method proposed by the FDA in 1973 for analysis of talc was an optical procedure as described below (1):

Weigh out 1 milligram of a representative portion of talc on each of two microscope slides. Mix the talc with a needle on one slide with a drop of 1.574 refractive index liquid, and then the other with 1.590 liquid, and place on each a square or rectangular cover glass sufficiently large so that the liquid will not run out from the edge (ca. 18 mm square) and will provide a uniform particle distribution. Fibers counted by this method should meet the following criteria: (i) Length to width ratio of 1 or greater (ii) length of 5 μ m or greater (iii) width of 5 μ m or less. Count and record the number of asbestos fibers in each 1 milligram as determined from a scan of both slides with a polarizing microscope at a magnification of approximately 400 \times . In the 1.574 refractive index liquid, chrysotile fibers with indices less than 1.5% in both extinction positions may be present; in the 1.590 refractive index liquid, all types of asbestos fibers with indices exceeding 1.590 in both extinction positions may be present. Check the extinction and sign of elongation for tentative identification. For specific identifications of asbestos fibers, make additional mounts in appropriate refractive index liquids, and refer to the optical crystallographic data in the table. A count of not more than 1000 amphibole types of asbestos and not more than 100 chrysotile asbestos fibers per milligram-slide constitutes the maximum limit for the presence of these asbestos fibers in talc. These limits assure a purity of at least 99.9 percent free of amphibole types of asbestos fibers and at least 99.99 percent free of chrysotile asbestos fibers.

The problem with the proposed method is that talc flakes are often oriented vertically or at a sufficient angle that they appear to be needles and thus must be tested for refractive index (Fig. 13). A typical number of such particles is five per field of view. This

Dr. Alice Blount, Heavy Liquid Separation Method, 1991

Environmental Health Perspectives –
Journal of National Institute of
Environmental Health Sciences (NIEHS),
part of U.S. Dept. of Health and Human
Services



National Institute of
Environmental
Health Sciences



Tab 13, Blount 1991 article

HEAVY LIQUID SEPARATION

Dr. Reynolds, Dartmouth College (1974) Heavy Liquid Separation Preparation Method



CONFIDENTIAL

MARCH, 1974

To: Windsor Minerals Inc., Windsor, Vermont 05089
From: R.C. Reynolds Jr., Department of Earth Sciences,
Dartmouth College, Hanover, New Hampshire 03755
Subject: Analysis of Talc Products and Ores for Asbestiform
Amphiboles

INTRODUCTION

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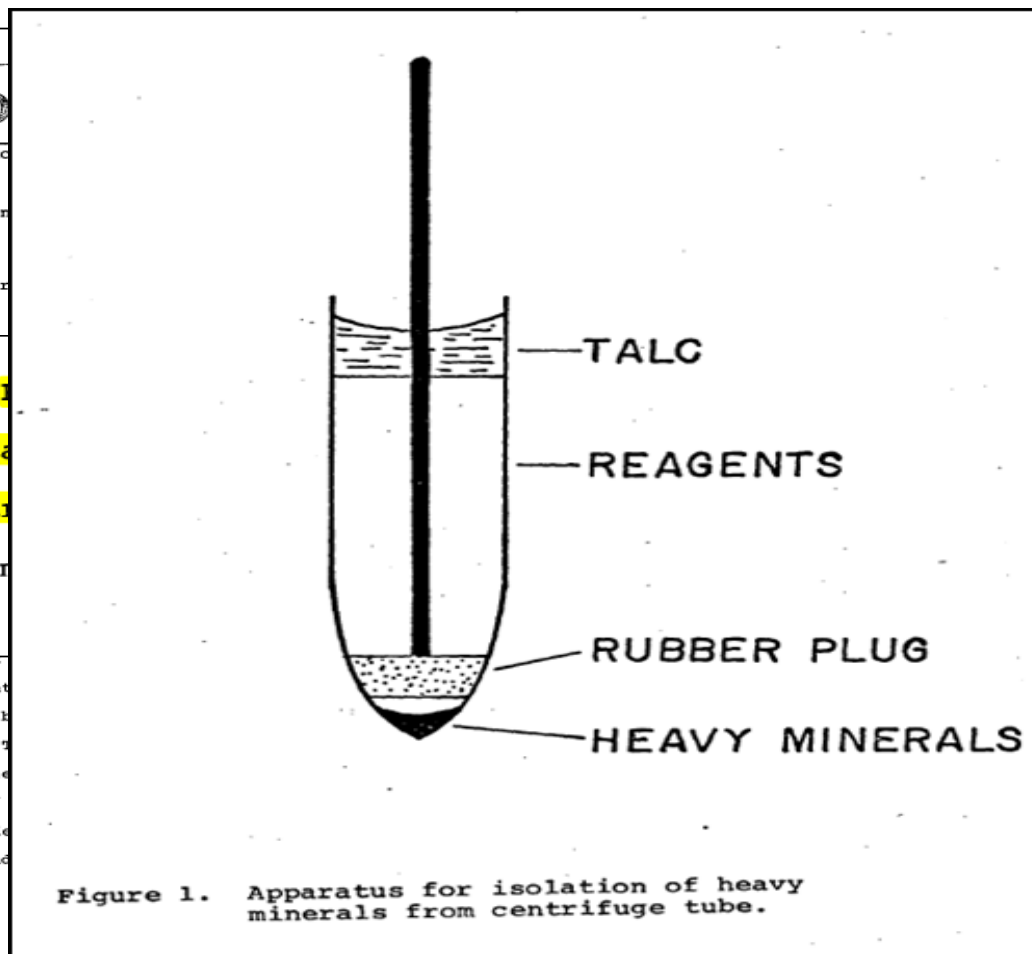
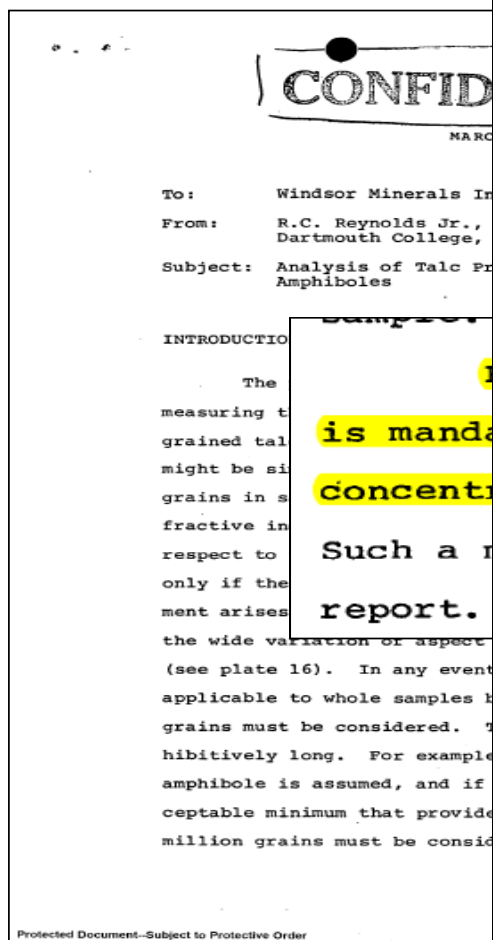
For the reasons described above, a concentration technique is mandatory because it brings the amphiboles into a reasonable concentration range for optical or other methods of analysis.

Such a method has been developed, and it is described in this report.

the wide variation of aspect ratios among the amphibole grains (see plate 16). In any event, grain-counting methods are inapplicable to whole samples because an inordinate number of grains must be considered. This makes the analysis time prohibitively long. For example, if a concentration of 100 ppm amphibole is assumed, and if 100 amphibole grains is the acceptable minimum that provides good statistical data, then one million grains must be considered. This requirement would be

Tab 17, 3/1974 Report from
Reynolds to Windsor Minerals,
Bates: JNJNL61_000029410,
JNJNL61_000029411,
JNJNL61_000029418

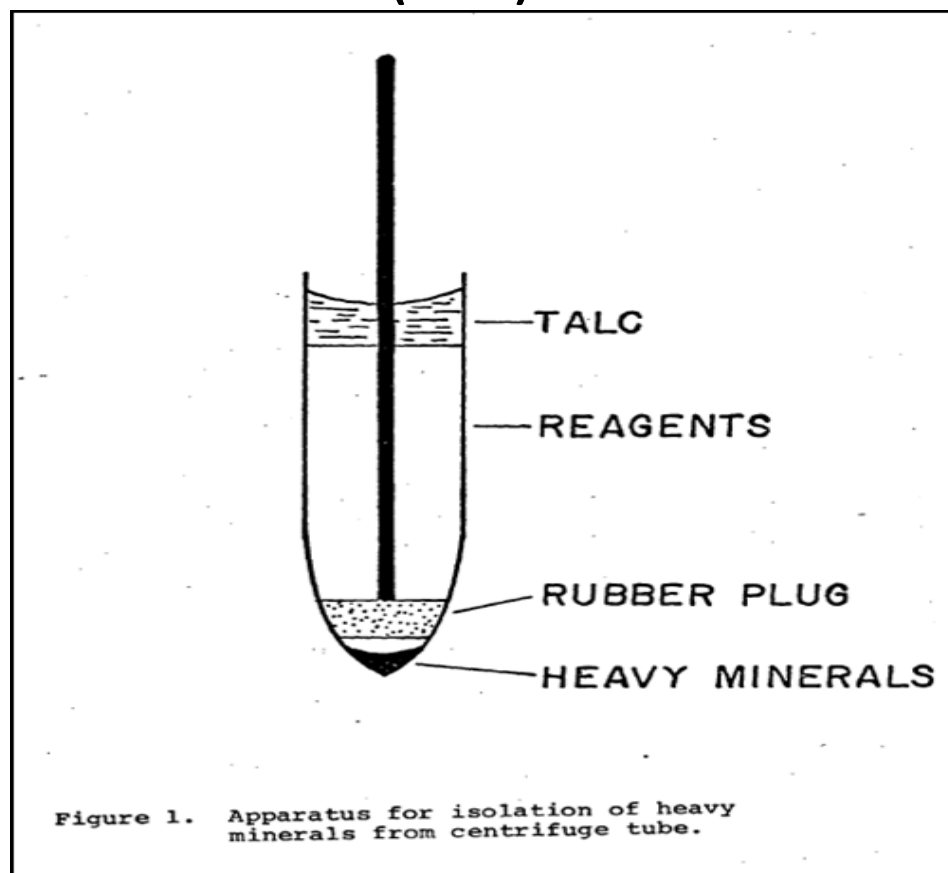
Dr. Reynolds, Dartmouth College (1974)



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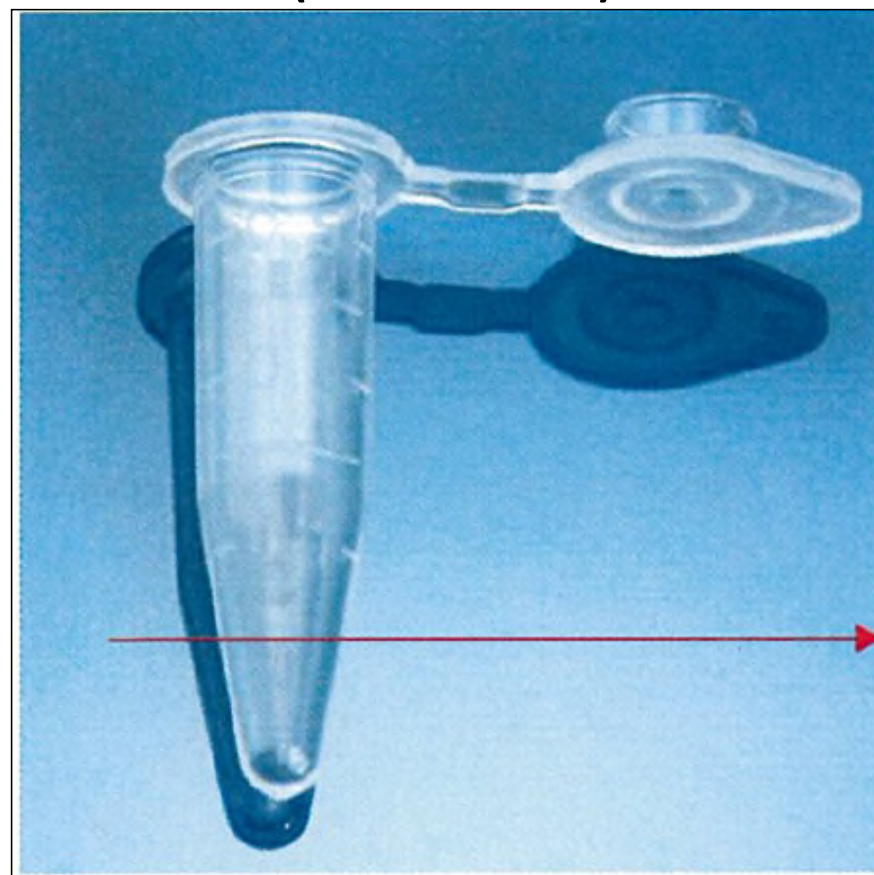
Tab 17, 3/1974 Report from
Reynolds to Windsor Minerals,
Bates: JNJNL61_000029410,
JNJNL61_000029411,
JNJNL61_000029418

**Dr. Reynolds, Dartmouth College
(1974)**



Tab 17, Bates: JNJNL61_000029418

**MAS
(2017-Present)**



Tab 9, Pg. 11

Dr. Reynolds, Dartmouth College (1974)

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Subject: A
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INTRODUCTION

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1. Mixtures of bromoform, methylene iodide, and benzethonium chloride monohydrate provide a suitable heavy liquid for the centrifugal separation of fiberform amphiboles from talc

2. The ore sample contains 2300 ppm actinolite, and the talc product contains ~170 ppm actinolite.

170 ppm = 0.017%

3. Actinolite is the dominant fiberform amphibole in the ore and talc product provided by Windsor Minerals. Small amounts of anthophyllite may be present.

LUG
NERALS

Dr. Reynolds, Dartmouth College (1974)

To: Windso
From: R.C. R
Dartmo
Subject: Analys
Amphib

INTRODUCTION:

The purpose of measuring the coefficient of grain growth might be simply to determine the number of grains in sample 16. The fractal index, D_f , with respect to talc, is only 1.5, which arises from the wide variation in grain size (see plate 16). This is not applicable to whole grains must be considerably long. Amphibole is acceptable minimum grain size is 100 microns.



Plate 7

Talc Ore, Acid Insoluble Heavy Fraction; x 100; n = 1.503
Actinolite, talc, chromite, and a large anthophyllite fiber.

GENTS

BER PLUG

VY MINERALS

heavy
tube.



Tab 18, ISO 22262-2, pg. v

ISO 22262-2 is specified for testing talc for asbestos

INTERNATIONAL STANDARD

ISO 22262-2

Tab 18, ISO 22262-2, pg. 1

This part of ISO 22262 specifies procedures for quantification of asbestos mass fractions below approximately 5 %, and quantitative determination of asbestos in vermiculite, other industrial minerals and commercial products that incorporate these minerals.

This part of ISO 22262 is applicable to the quantitative analysis of:

- a) any material for which the estimate of asbestos mass fraction obtained using ISO 22262-1 is deemed to be of insufficient precision to reliably classify the regulatory status of the material, or for which it is considered necessary to obtain further evidence to demonstrate the absence of asbestos;
- b) resilient floor tiles, asphaltic materials, roofing felts and any other materials in which asbestos is embedded in an organic matrix;
- c) wall and ceiling plasters, with or without aggregate;
- d) mineral products such as wollastonite, dolomite, calcite, talc or vermiculite, and commercial products containing these minerals.

ISO 22262-2 is specified for testing talc for asbestos

Product	Examples of application	Typical asbestos type and mass fraction if asbestos is present	Analysis in accordance with	Optimum analytical procedure
Talc (asbestos content dependent on deposit)	<ul style="list-style-type: none"> – Release agents for electric cables, rubber products – Release agents in the confectionery industry – Tailor's chalk – Paper manufacture – Medicine, cosmetics 	Chrysotile and/or actinolite/tremolite	ISO 22262-2	<p>Talc is not amenable to gravimetric matrix reduction methods.</p> <p>For chrysotile, preparation of TEM specimens from the untreated material is the optimum procedure, followed by examination by the mass counting procedure.</p> <p>For amphibole, either centrifugation in heavy liquid, followed by evaluation of the centrifugate by microscopy, or preparation of TEM specimens from the untreated material is the optimum procedure, followed by examination using the mass counting procedure.</p>

INTERNATIONAL
STANDARD

ISO
22262-2

Tab 18, ISO 22262-2 Pg. 38

16.3 Determination of amphibole in talc

INTERNATIONAL
STANDARD

ISO
22262-2

Centrifugation in a heavy liquid can permit separation of amphibole from talc, because the published density range for talc is 2 580 kg/m³ to 2 830 kg/m³, and the minimum density for the amphibole asbestos varieties is 3 000 kg/m³. Separation of amphibole from talc is therefore possible using a liquid of density 2 850 kg/m³. Calculate the required centrifugation time according to Annex B. Use the centrifugation method described in 15.4.3. Identify any asbestiform amphibole in the centrifugate according to the procedures specified in ISO 22262-1. Quantify any asbestiform amphibole in the centrifugate by the

Tab 18, ISO 22262-2 Pg. 29-30

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ATEM-ISO 22262-2 TEM Sample Preparation



Density Separation

Tab 9, MAS 1/15/19 Report, Pg. 10

Approximately 20 to 30 mg (Sartorius Research Balance) from the muffled talc sample aliquot was placed into a labeled Eppendorf micro-centrifuge tube (MCT) (Premium 1.5mL MCT Graduated Tubes Cat. No. 05-408-12). Approximately 1.2 ml of Heavy Liquid (Lithium heteropolytungstates solution, GeoLiquids, Inc., Cat. No. LST010 density 2.85 g/cc) was added to the MCT containing the talc samples prepped and mixed with a disposable mixing rod for

Analytical Tool / Analysis Method (TEM, PLM)

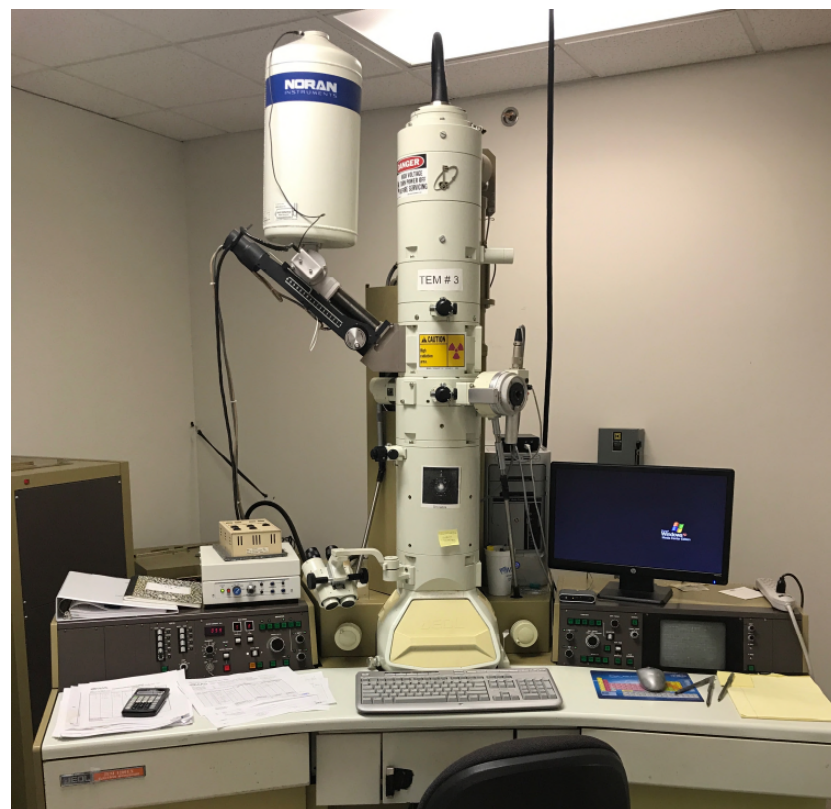
Analytical Tools (Microscopes)



Polarized Light Microscope (PLM)



Transmission Electron Microscope (TEM)



ISO 22262-2 – After Heavy Liquid Separation, PLM, SEM or TEM

ISO 22262-2:2014(E)

INTERNATIONAL
STANDARD

ISO
22262-2



8 Principle

A known weight of the material is heated in a furnace to a temperature of $450\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ to remove organic materials. Depending on the nature of the sample, the residue from the heating is treated with either hydrochloric or sulphuric acid to dissolve acid-soluble constituents. If appropriate, water sedimentation is then used to separate aggregate fragments and particles. For sensitive quantification of amphibole, some materials may require a refluxing treatment in acid, followed by a reflux treatment in sodium hydroxide. Alternatively, amphibole can be separated from many other constituents of lower densities by centrifugation in a heavy liquid. The proportion of asbestos in the residue from these treatments is then determined by appropriate PLM, SEM or TEM techniques.

TEM Analytical Procedure: 3 Steps

ATEM Amphibole Analysis Procedure

JEOL 1200EX ATEMs equipped with either a Noran or an Advanced Analysis Technologies (light element) energy dispersive x-ray analyzer (EDXA) were employed for this analysis. ATEM samples were analyzed at a screen magnification of 20,000X. Amphibole fibers or bundles with substantially parallel sides and an aspect ratio of 5:1 or greater, and at least 0.5µm in length were counted as regulated asbestos fibers and bundles per standard TEM counting rules as described by ASTM D5755, ASTM D5756, ISO 10312, ISO 13794, AHERA (TEM section only) and D7712-11.^{10,11,12,13,14,15}

#2

Positive identification of amphibole asbestos requires EDXA for mineral chemistry confirmation and selected area electron diffraction (SAED) for each amphibole type. At times, amphibole

#3

Tab 9, MAS 1/15/19 Report, Pg. 12.

TEM Analytical Procedure: 3 Steps

[CANCER RESEARCH 55, 2232-2235, June 1, 1995]

Advances in Brief

Crocidolite Asbestos Fibers in Smoke from Original Kent Cigarettes¹

William E. Longo, Mark W. Rigler,² and John Slade

Materials Analytical Services, Inc., Norcross, Georgia 30092 [W. E. L., M. W. R.], and Department of Medicine, University of Medicine and Dentistry of New Jersey and St. Peter's Medical Center, New Brunswick, New Jersey 08901 [J. S.]

Puff residue was examined for asbestos structures containing **#1** in the use of either a JEOL 1200 EX or Hitachi 7110 TEM at a magnification of $\times 20,000$. Asbestos structures were identified positively by their morphology, by their chemistry with the use of a Tracor Northern TN 5500 or Kevex Delta Class 5

#2 energy dispersive X-ray analysis system, and by their crystalline structure with the use of selected area electron diffraction. Asbestos structures were counted and classified according to standard EPA protocols (11).

#3

11. Environmental Protection Agency. Asbestos Containing Materials in Schools: Final Rule and Notice. 40 CFR Part 763, p. 41864. Washington DC: Federal Register III, 1987.

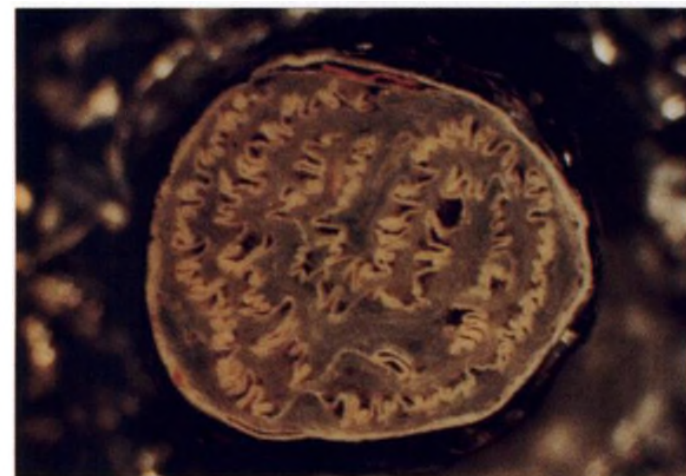
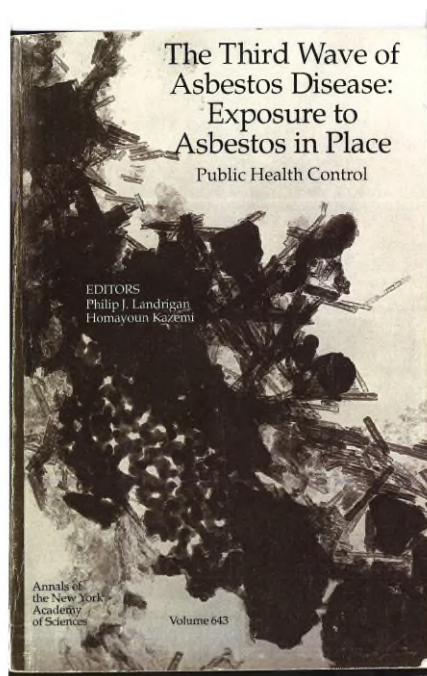


Fig. 1. Mouthpiece end of an original Kent Micronite filter.

Tab 10, Longo et al. 1995, Pgs. 2232, 2233, 2235

TEM Analytical Procedure: 3 Steps



Mineral Fiber Content of Lung Tissue in Patients with Environmental Exposures: Household Contacts vs. Building Occupants

VICTOR L. ROGGLI^a AND WILLIAM E. LONGO^b

^a*Department of Pathology
Durham Veterans Administration, and
Duke University Medical Centers
Durham, North Carolina 27710*

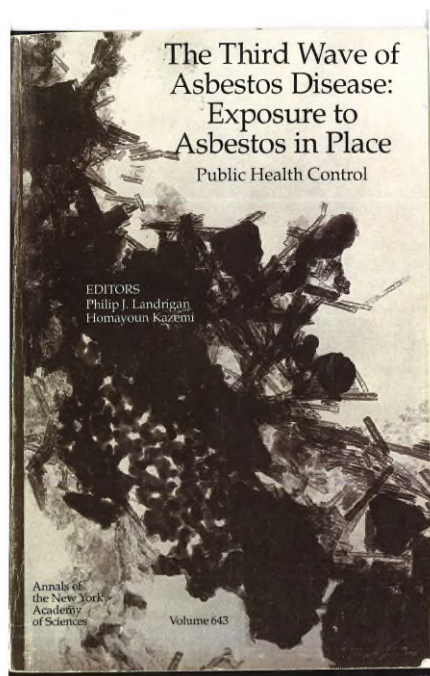
^b*Materials Analytical Services, Inc.
Norcross, Georgia 30092*

fibers were analyzed per case and classified as asbestiform (amosite, crocidolite, tremolite, anthophyllite, actinolite, or chrysotile) or nonasbestiform on the basis of morphology and chemical composition as previously described.^{15,16}

Additional studies were performed in one case (Case 8, TABLE 2) to further characterize the mineral content of lung tissue. Paraffin-embedded lung parenchyma was deparaffinized in xylene (three changes, 2 hours each) and ashed in a low-temperature plasma asher for 100 hours. The dry weight of four combined specimens in this case was 0.18 gram. After ashing was complete, the remaining residue was suspended in 24 ml of filtered, deionized water and then sonicated for 10 minutes. The suspension was then filtered through a 0.45 μ -pore-sized mixed cellulose ester filter, which was then prepared by the direct method for examination by transmission electron microscopy, selected area electron diffraction, and energy-dispersive spectrometry (TEM/SAED/EDS).¹⁹ Also examined with the

Tab 10A, Third Wave of Asbestos Disease, 1991, Pg. 511, 513

TEM Analytical Procedure: 3 Steps



Mineral Fiber Content of Lung Tissue in Patients with Environmental Exposures: Household Contacts vs. Building Occupants

VICTOR L. ROGGLI^a AND WILLIAM E. LONGO^b

^aDepartment of Pathology
Durham Veterans Administration, and
Duke University Medical Centers
Durham, North Carolina 27710

^bMaterials Analytical Services, Inc.
Norcross, Georgia 30092

In addition, a plaster sample was obtained from the high school where case 8 was employed and was analyzed for its mineral content by means of polarized light microscopy with dispersion staining²⁰ and by TEM/SAED/EDS. Also, the

about 20% of cases according to the study by McDonald *et al.*²⁶ Since multiple components of the acoustical ceiling plaster from the building in which this patient worked were also found in her lung tissue samples, this is the most likely source of the tremolite asbestos fibers that were identified. There was no evidence of exposure to cosmetic talc and no evidence of household exposure on the basis of her husband's occupational history. Furthermore, the presence of histologically

Tab 10A, Third Wave of Asbestos Disease, 1991, Pg. 511, 513, 517

TEM Analytical Procedure: Identification of Asbestos as a Contaminant

Zonolite Attic Insulation Exposure Studies

WILLIAM M. EWING, STEVE M. HAYS, RICHARD HATFIELD, WILLIAM E. LONGO,
JAMES R. MILLETTE

INT J OCCUP ENVIRON HEALTH 2010;16:279-290

Sampling Methods

Air, dust, and bulk samples were collected as part of this study. Sample logs and chain-of-custody forms were completed for all samples. Air, dust, and bulk samples were stored and transported separately to minimize the opportunity of cross-contamination between samples.

The amphibole asbestos species identified by electron microscopy or polarized light microscopy in air, dust, or bulk samples are reported herein as “Libby amphiboles” and consisted of fibrous tremolite, richterite, winchite, and actinolite.^{11,12}



Figure 5—View of attic in home C.

Tab 12, Longo et al. 2010, Pgs. 279,
281, 282

Zonolite Attic Insulation Exposure Studies

WILLIAM M. EWING, STEVE M. HAYS, RICHARD HATFIELD, WILLIAM E. LONGO,
JAMES R. MILLETTE

INT J OCCUP ENVIRON HEALTH 2010;16:279-290

All air samples were submitted to a laboratory accredited by the American Industrial Hygiene Association (AIHA) and the National Voluntary Laboratory Accreditation Program (NVLAP) (administered by the National Institute of Standards and Technology (NIST), or were A2LA accredited under ISO Standard 17025. Personal air samples collected on 0.8 μm pore size MCE filters were analyzed by phase contrast microscopy (PCM) as described in NIOSH method 7400.¹³ Personal and area air samples collected on 0.45 μm MCE filters were analyzed by transmission electron microscopy (TEM) using the direct preparation techniques described in the EPA Code of Federal Regulations.¹⁴ This method is commonly referred to as the EPA AHERA method. The results of the PCM samples

Zonolite Attic Insulation Exposure Studies

WILLIAM M. EWING, STEVE M. HAYS, RICHARD HATFIELD, WILLIAM E. LONGO,
JAMES R. MILLETTE

INT J OCCUP ENVIRON HEALTH 2010;16:279-290

All air samples were submitted to a laboratory accredited by the American Industrial Hygiene Association (AIHA) and the National Voluntary Accreditation Program (NVLAP) (administered by the National Institute of Standards and Technology (NIST), or were A2LA accredited under ISO 17025. Personal air samples collected on 37 mm size MCE filters were analyzed by phase contrast microscopy (PCM) as described in NIOSH Method 7400.¹³ Personal and area air samples collected on 37 mm MCE filters were analyzed by transmission electron microscopy (TEM) using the direct preparation techniques described in the EPA Code of Federal Regulations.¹⁴ This method is commonly referred to as the EPA AHERA method. The results of the PCM samples

Analyses conducted of the bulk ZAI in these homes and other buildings generally results in amphibole asbestos concentrations of less than 1% and often less than 0.1 %. However, the exposure data presented here, and the exposure data from the Manitoba building referenced earlier, demonstrate that significant exposures can still occur. These exposures can be in excess of current regulatory exposure limits.

ASTM D5755 – 3 Steps



Designation: D5755 – 09 (Reapproved 2014)^{e1}

Standard Test Method for
Microvacuum Sampling and Indirect Analysis of Dust by
Transmission Electron Microscopy for Asbestos Structure
Number Surface Loading¹

#1

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

#3

#2

AHERA: TEM Method developed by “committee of leading microscopists”

requirement will be phased in gradually. EPA convened a committee of leading microscopists from private and Federal laboratories to produce an analytical protocol specific for post-abatement clearance monitoring. Each microscopist had extensive experience in TEM, scanning electron microscopy (SEM), and airborne asbestos analysis. The unanimous conclusion of the microscopists was that, for purposes of clearance air monitoring, TEM was the technique of choice. Consequently, an

EPA chose to require analysis by TEM for four reasons: (1) TEM is capable of measuring the smallest diameter fibers; (2) based on existing, validated methods, a formal protocol has been developed; (3) TEM has been validated by intra- and inter-laboratory comparisons conducted by NBS; and (4) a formal laboratory accreditation program for TEM laboratories is currently under development by the NBS.

Step 1: Morphology – AHERA Method

F. TEM Method

9. *Recording Rules.*

a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 μm is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any

i. *Fiber.* A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle.* A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

≥ 5:1 Aspect Ratio
≥ 0.5 μ in length



Why Does the EPA Use a 5:1 Aspect Ratio?

Regarding the use of TEM, several commenters suggested that the aspect ratio (length to width) should be extended to 10:1. For the purpose of TEM measurement by the methods in Appendix A, any elongated particle having a minimum length of 0.5 μm , parallel sides, and an aspect ratio

(length to width) of 5:1 or larger is defined as a fiber. This represents a change in the previous EPA proposed TEM methodologies which examine fibers with aspect ratios of 3:1 and above; it follows the direction set by NIOSH in proposing modified counting rules in the 7400 method. It is consistent with the panel of microscopists' observations that asbestos structures have aspect ratios equal to and greater than 5:1 whereas the majority of nonasbestos structures, minerals and particles, for example, gypsum, have aspect ratios of less than 5:1. Analysis of these nonasbestos structures tends to comprise a large portion of the time required for sample analysis. EPA believes that further research is needed to justify the extension of aspect ratio to 10:1. Consequently, for the purpose of TEM building clearance, fibers must have an aspect ratio of at least 5:1.

$\geq 5:1$ = asbestos

$< 5:1$ = majority non-asbestos



Tab 20, Federal Register, Vol. 52, No. 210, October 30, 1987, Pg. 41840

Peer-Reviewed Scientific Literature Supports Use of 5:1 Aspect Ratio



Tab 22, Millette
2015, Pgs. 11,
16

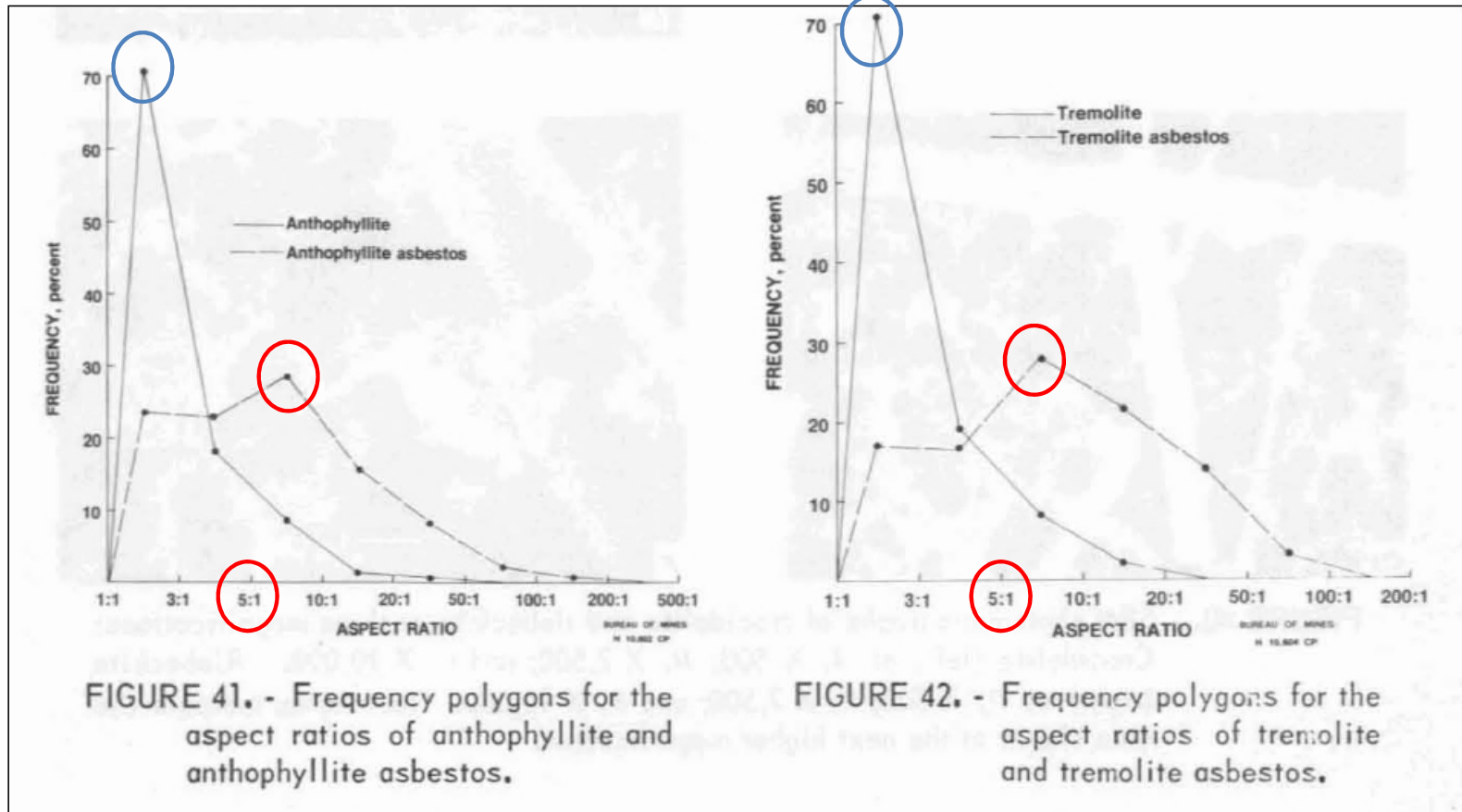
THE MICROSCOPE • Vol. 63:1, pp 11–20 (2015)

Procedure for the Analysis of Talc for Asbestos

James R. Millette, Ph.D., D-IBFES
Millette Technical Consulting¹

ther way. Research by Wylie (25) reported in 1985 showed that 50% of the fibers in a known amosite (grunerite) asbestos sample would not be counted if a 20:1 aspect ratio were used as a criterion. Comparison of the aspect ratio plots in the 1977 Bureau of Mines Circular (26) shows that a criterion of about 5:1 aspect ratio appears to be the best aspect ratio discriminator for asbestos versus non-asbestos fibers. The 5:1 aspect ratio is used in AHERA; ASTM methods D6281, D5755, D5756 and D6480; and ISO 10312 and 13794.

Campbell, Bureau of Mines, 1977



Campbell, Bureau of Mines, 1977

ratio. Cleavage fragments will generally have a frequency maximum less than 3 to 1, whereas the asbestiform varieties will fall between 10 to 1 and 20 to 1 or higher, depending on the characteristics of the mineral and the history of the sample, particularly the type and degree of milling. If any shape or size limits are placed on characterizing mineral particulates, such limits should be based on medical evidence or on some limitation of the characterizing technique and so stated.

“Cleavage fragments” – “generally...less than 3 to 1” aspect ratio

ASTM D5755 – Step 1: Morphology



Designation: D5755 – 09 (Reapproved 2014)^{e1}

**Standard Test Method for
Microvacuum Sampling and Indirect Analysis of Dust by
Transmission Electron Microscopy for Asbestos Structure
Number Surface Loading¹**

3.2.6 *fiber*—a structure having a minimum length of 0.5 μm , an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

≥ 5:1 Aspect Ratio
≥ 0.5 μ in length

ISO 22262 – Step 1: Morphology



INTERNATIONAL
STANDARD

ISO
22262-1

First edition
2012-07-01

INTERNATIONAL
STANDARD

ISO
22262-2

2.28 Tab 25, ISO 22262-1, Pg. 5

fibre
elongated particle which has **parallel or stepped sides**

[ISO 13794:1999,^[4] 2.26]

NOTE For the purposes of this part of ISO 22262, a fibre is defined to have an aspect ratio greater than or equal to 3:1.

Tab 18, ISO 22262-2, Pg. 4, Sec. 3.19 = SAME

INTERNATIONAL
STANDARD

ISO
13794

First edition
1999-07-15

2.26 Tab 25A, ISO 13794, Pg. 4

fibre
elongated particle which has **parallel or stepped sides**

NOTE For the purposes of this International Standard, a fibre is defined to have an **aspect ratio equal to or greater than 5:1**
and a minimum length of 0,5 µm.

≥ 5:1 Aspect Ratio
≥ 0.5 µ in length

MAS TEM Analysis of J&J Talc Products – Application of 3 Step Analytical Protocol

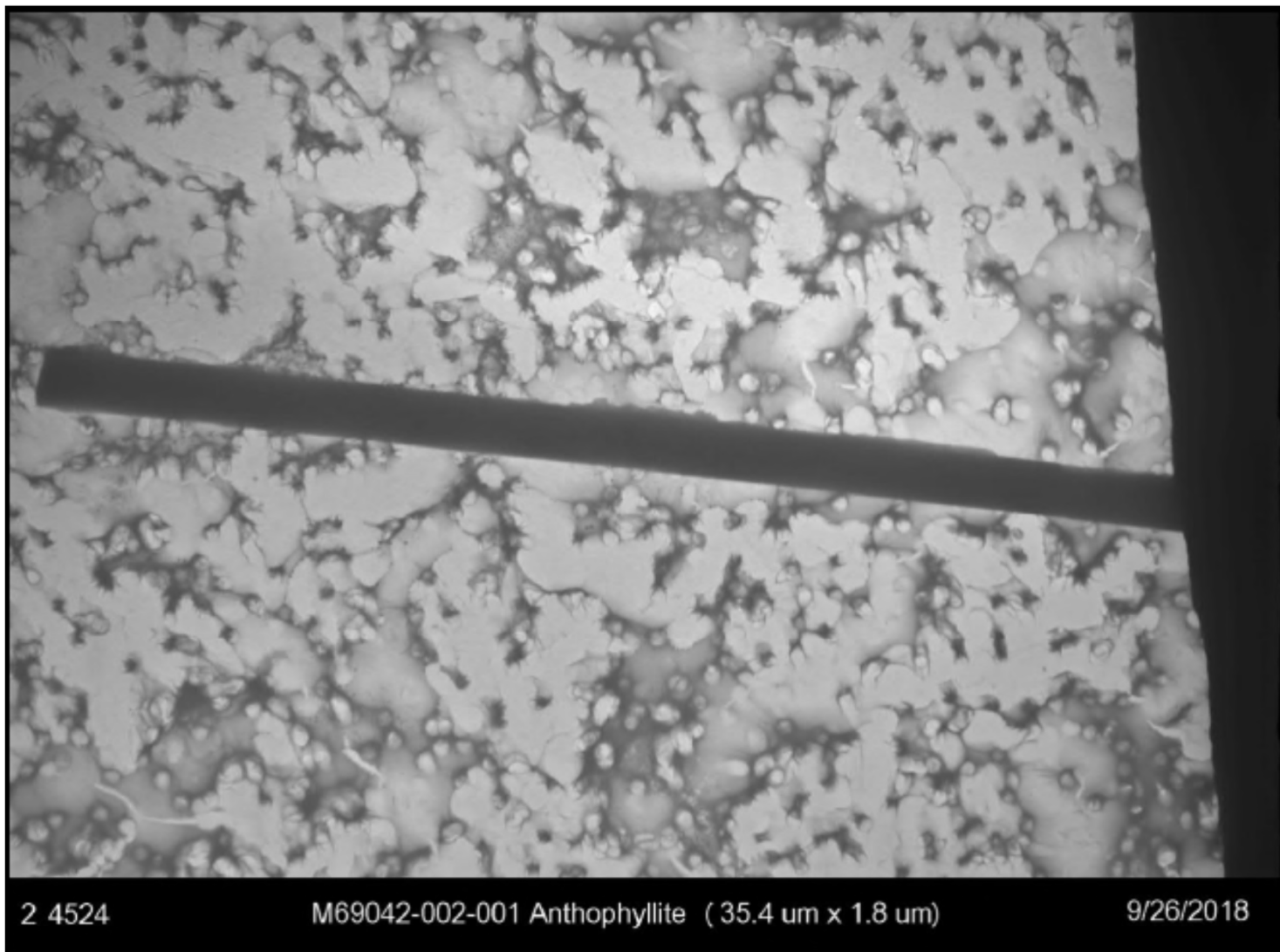
Example 1
Sample M69042-002
1978 JBP Sample



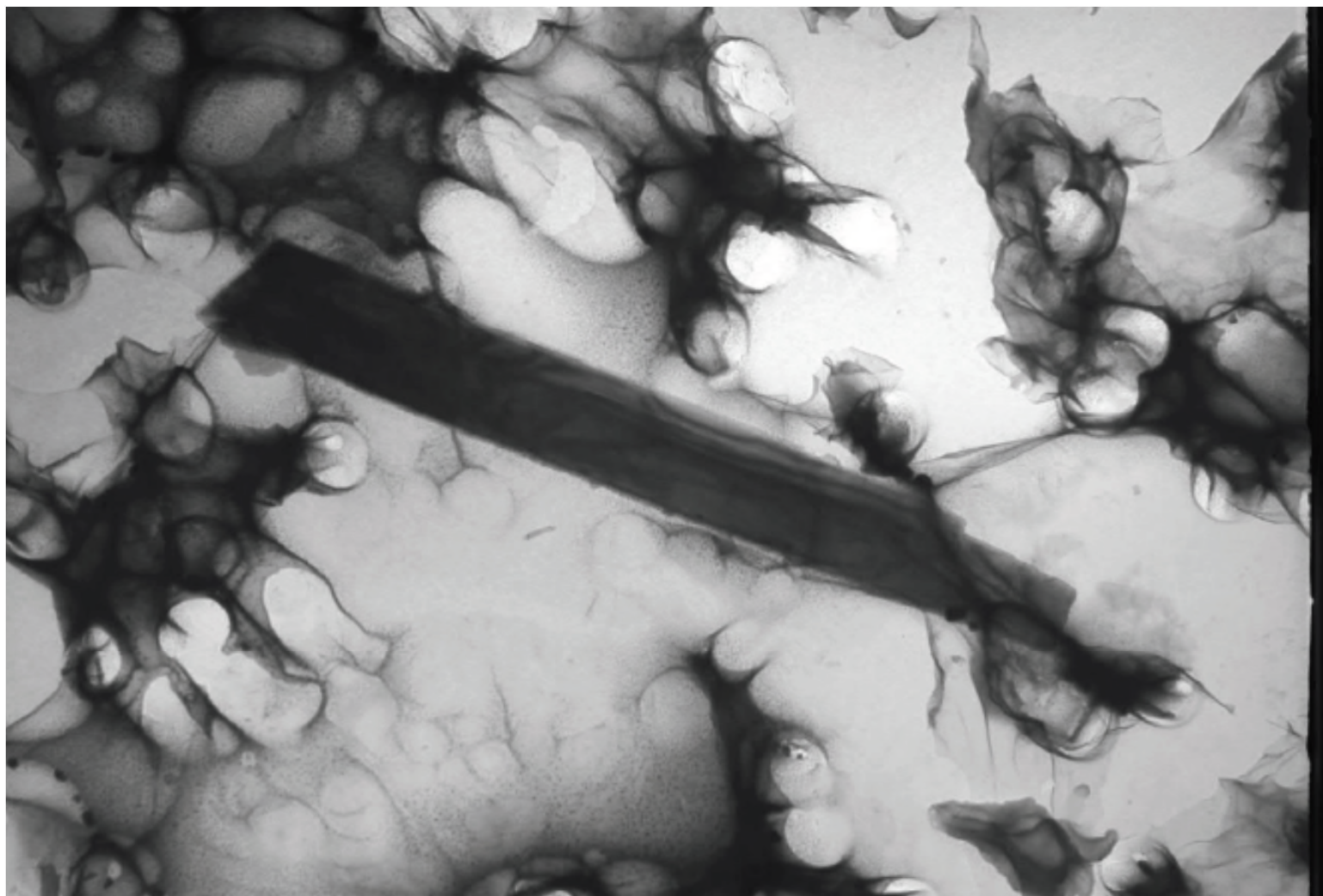


Step 1: Morphology

Length: 35.4 μm
Substantially Parallel Sides
Aspect Ratio - 19.7 to 1



MAS 1/15/19 Report Backup
Data Binder,
Bates : Longo-MDL_00881,
Tab 9A



Step 1: Morphology

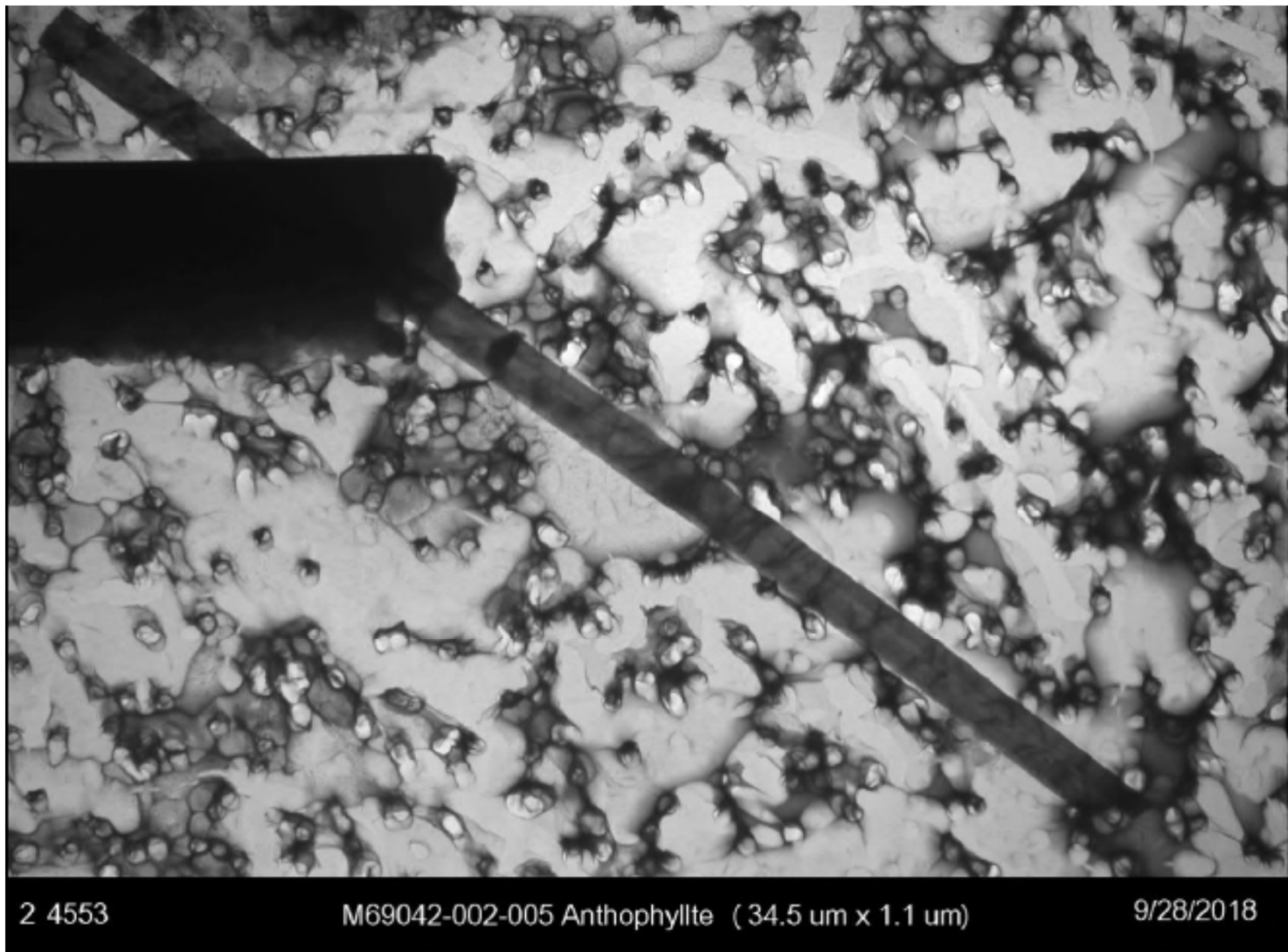
Length: 6 μm
Substantially Parallel Sides
Aspect Ratio - 8.6 to 1

MAS 1/15/19 Report
Backup Data Binder,
Bates: Longo-MDL_00893,
Tab 9A

2 4546

M69042-002-004 Anthophyllite (6.0 μm x 0.7 μm)

9/27/2018



Step 1: Morphology

Length: 34.5 μm
Substantially Parallel Sides
Aspect Ratio – 31.4 to 1

MAS 1/15/19 Report
Backup Data Binder,
Bates: Longo-MDL_00897,
Tab 9A

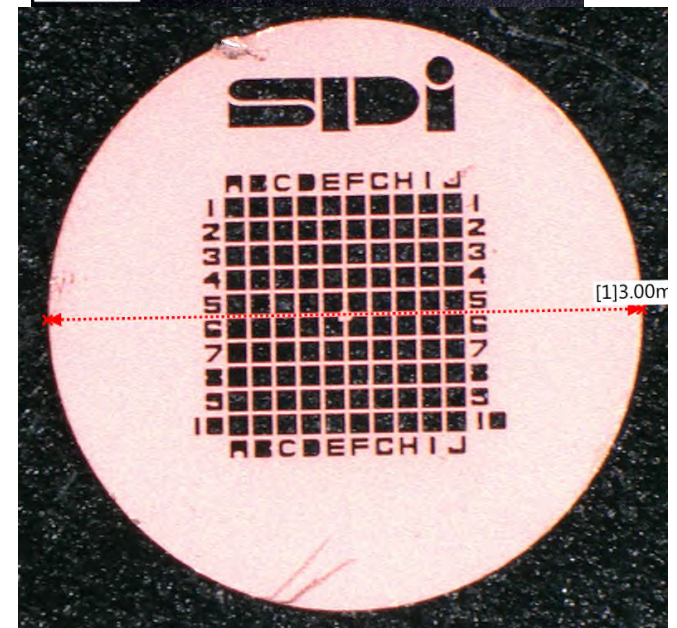
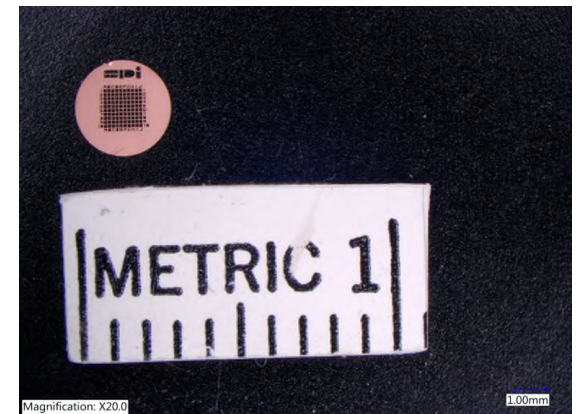
2 4553

M69042-002-005 Anthophyllite (34.5 μm x 1.1 μm)

9/28/2018

TEM Bulk Talc Structure Count Sheet					
Project/ Sample No.	M69042-002		Grid Box #	8621	No. of Grids Counted
Analyst	Anthony Keeton		Length	Width	G. O. Area
Date of Analysis	9/26/2018 - 9/28/2018 & 10/27/2018		105	105	11025
Initial Weight(g)	0.02000		105	105	11025
Analysis Type	Post Separation Talc Analysis		Grid Acceptance	Yes	Average
Scope No.	Accelerating Voltage	100 KV	Loading%	12%	G.O.s Counted
2	Screen Magnification	20 KX	Area Examined mm ²		1.103

Str. #	Grid Opening	Structure	Asbestos Type	Length	Width	Ratio	SAED	EDS
NSD	B2-B6							
NSD	B7							
1	B8	Bundle	Anthophyllite	35.4	1.8	19.7	X	X
2		Bundle	Anthophyllite	12.4	1.1	11.3	X	X
NSD	B9							
NSD	B10							
NSD	C3							
NSD	C4							
NSD	C5							
NSD	C6							
NSD	C7							
NSD	C8							
NSD	C9							
NSD	C10							
3	E1	Bundle	Anthophyllite	6.4	1.1	5.8	X	X
NSD	E2							
NSD	E3							
NSD	E4							
NSD	E5							
NSD	E6							
NSD	E7							
NSD	E8							
4	E9	Bundle	Anthophyllite	6	0.7	8.6	X	X
NSD	E10							
5	H7	Bundle	Anthophyllite	34.5	1.1	31.4	X	X



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00875, Tab 9A

Aspect Ratio

M69042-002

Str. #	Length (μm)	Width (μm)	Aspect Ratio	Structure Type	Asbestos Type
-1	35.4	1.8	19.7	Bundle	Anthophyllite
-2	12.4	1.1	11.3	Bundle	Anthophyllite
-3	6.4	1.1	5.8	Bundle	Anthophyllite
-4	6.0	0.7	8.6	Bundle	Anthophyllite
-5	34.5	1.1	31.4	Bundle	Anthophyllite
-6	11.5	1.2	9.6	Bundle	Anthophyllite
-7	11.5	1.0	11.5	Bundle	Anthophyllite

Average Aspect Ratio: 14.0

Step 2: EDS/EDXA – AHERA Method

f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70 s/mm² concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)





Step 2: EDS/EDXA – AHERA Method



j. Energy Dispersive X-Ray Analysis (EDXA).

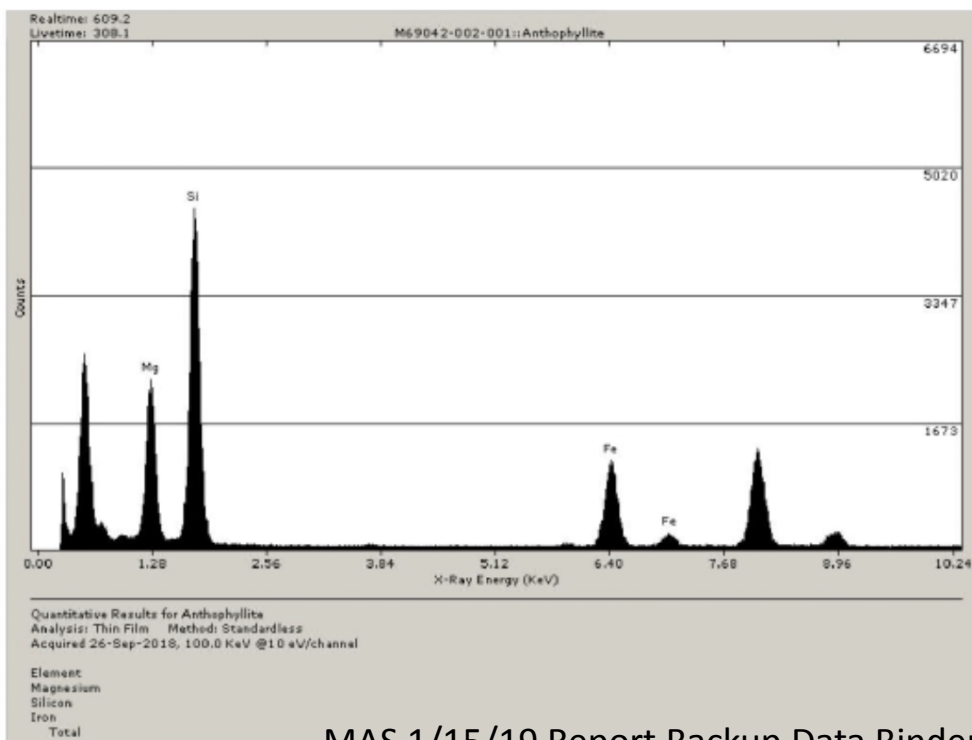
i. Required of all amphiboles which would cause the analysis results to exceed the 70 s/mm² concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

ii. Can be used alone to confirm chrysotile after the 70 s/mm² concentration has been exceeded.

iii. Can be used alone to confirm all non-asbestos.

iv. Compare spectrum profiles with profiles obtained from asbestos standards. The closest match identifies and categorizes the structure.

Tab 21, EPA AHERA, Pg. 893



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00878, Tab 9A

Standard Methodology: Compare “EDXA Spectra” with “Reference Spectra”

c. Calibration of the EDXA System. Initially, the EDXA system must be calibrated by using two reference elements to calibrate the energy scale of the instrument. When this has been completed in accordance with the manufacturer's instructions, calibration in terms of the different types of asbestos can proceed. The EDXA detectors vary in both solid angle of detection and in window thickness. Therefore, at a particular accelerating voltage in use on the TEM, the count rate obtained from specific dimensions of fiber will vary both in absolute X-ray count rate and in the relative X-ray peak heights for different elements. Only a few minerals are relevant for asbestos abatement work, and in this procedure the calibration is specified in terms of a “fingerprint” technique. The EDXA spectra must be recorded from individual fibers of the relevant minerals, and identifications are made on the basis of semiquantitative comparisons with these reference spectra.



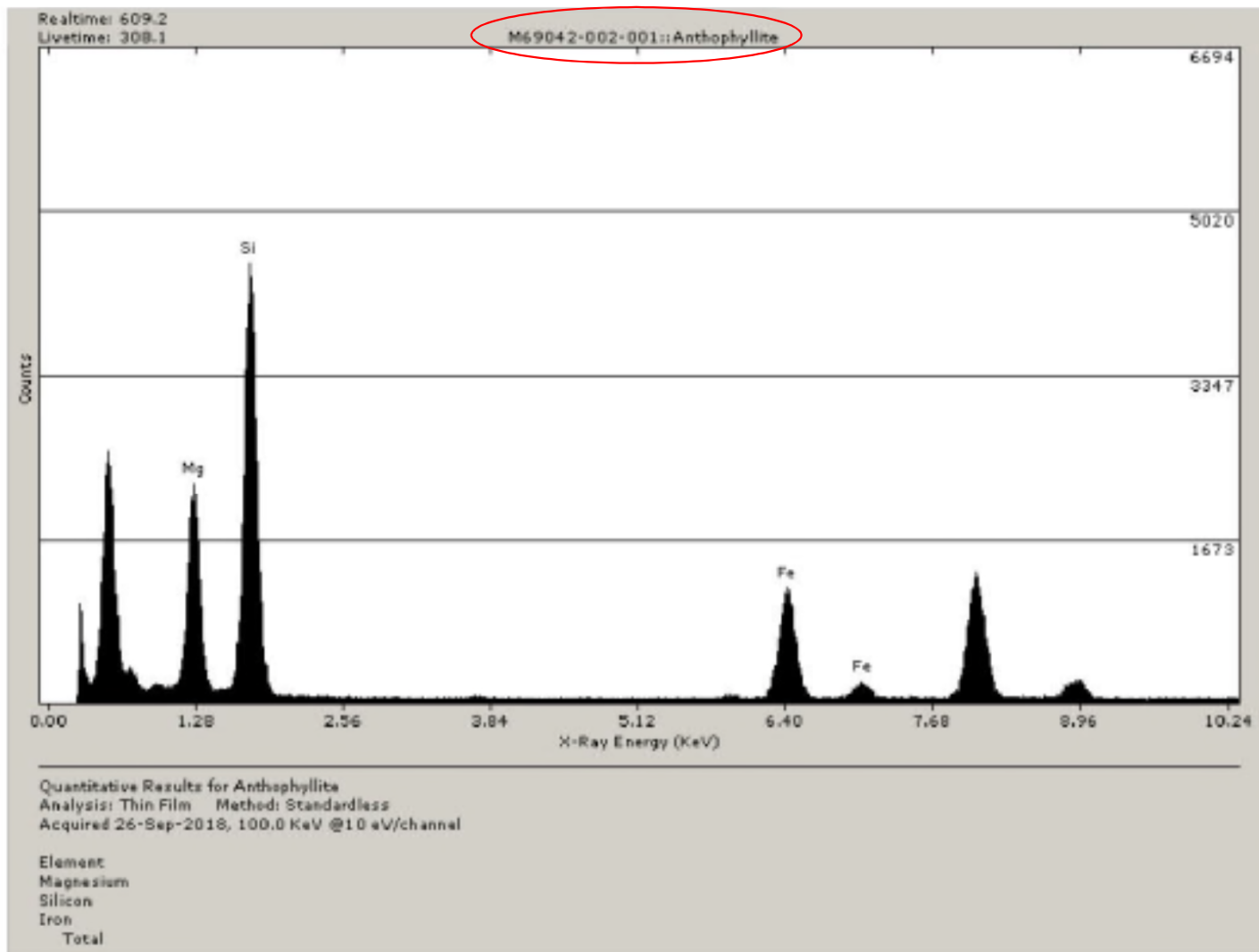
ASTM D5755 – Step 2: EDXA



Designation: D5755 – 09 (Reapproved 2014)^{e1}

**Standard Test Method for
Microvacuum Sampling and Indirect Analysis of Dust by
Transmission Electron Microscopy for Asbestos Structure
Number Surface Loading¹**

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one **X-ray spectrum** for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.



TEM - EDS/EDXA

M69042-002-001

Step 2: Chemistry by EDXA

Consistent with Anthophyllite
Asbestos

MAS 1/15/19 Report Backup Data
Binder, Bates: Longo-MDL_00878,
Tab 9A

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STANDARD

ISO
22262-1

First edition
2012-07-01



9.5 Qualitative analysis by TEM

9.5.7 Anthophyllite

Classify a fibre as anthophyllite if:

- a) the fibre is straight and exhibits no evidence of a ribbon-like structure;
- b) the **Mg and Si peaks are comparable in ratio to those of reference anthophyllite** — anthophyllite from some sources may not exhibit a peak from Fe, although in commercial anthophyllite a peak from Fe will probably be observed;
- c) no statistically significant peaks from Na or Al are present;
- d) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

Step 3: SAED – AHERA Method

d. Visual identification of electron diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm² concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)



Step 3: SAED – AHERA Method

- i. Center the structure, focus, and obtain an ED pattern. (See Microscope Instruction Manual for more detailed instructions.)
- ii. From a visual examination of the ED pattern, obtained with a short camera length, classify the observed structure as belonging to one of the following classifications: chrysotile, amphibole, or nonasbestos.



Step 3: SAED – AHERA Method

13. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions.



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2012-07-01



Annex F (normative)

Asbestos identification by TEM in commercial materials

F.1 General

For the identification of asbestos in some types of bulk materials, particularly for those in which PLM examination yields ambiguous results, TEM examination can usually resolve the ambiguities and provide definitive identification of the fibres. In most cases, acquisition of an EDXA spectrum provides sufficient evidence to identify any of the asbestos varieties. Discrimination between talc and anthophyllite, however, cannot be reliably achieved on the basis of an EDXA spectrum alone, because the chemical compositions of the two minerals are very similar. Electron diffraction permits discrimination between talc and anthophyllite on the basis of their different crystal structures.

ISO 22262-1: SAED

ED patterns can be particularly useful for differentiating fibrous talc from anthophyllite asbestos, both of which have similar EDXA spectra. ED of talc produces a pseudo-hexagonal pattern that does not change as the fibre is tilted using the goniometer. Anthophyllite asbestos, on the other hand, produces assorted spots appearing and disappearing along layer lines as the fibre is tilted using the goniometer. ED patterns can also be a useful diagnostic tool for chrysotile that is so heavily coated with matrix that EDXA is inconclusive. Detection of the 002, 110, and 130 reflections as shown in Figure F.12 in conjunction with 0,53 nm layer-line spacing confirms the presence of chrysotile.

INTERNATIONAL
STANDARD

ISO
22262-1

First edition
2012-07-01



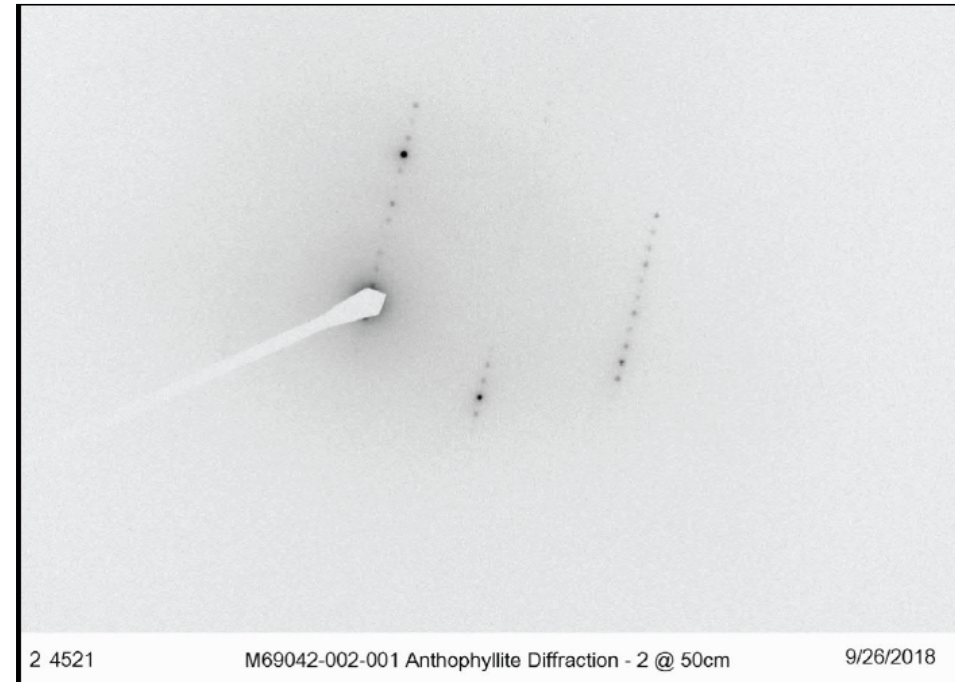
TEM - SAED

M69042-002-001

Step 3: Crystal Structure by SAED

Result: Anthophyllite Asbestos

Zero-Tilt Diffraction Pattern



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00879-880, Tab 9A

ASTM D5755 – Step 3: SAED

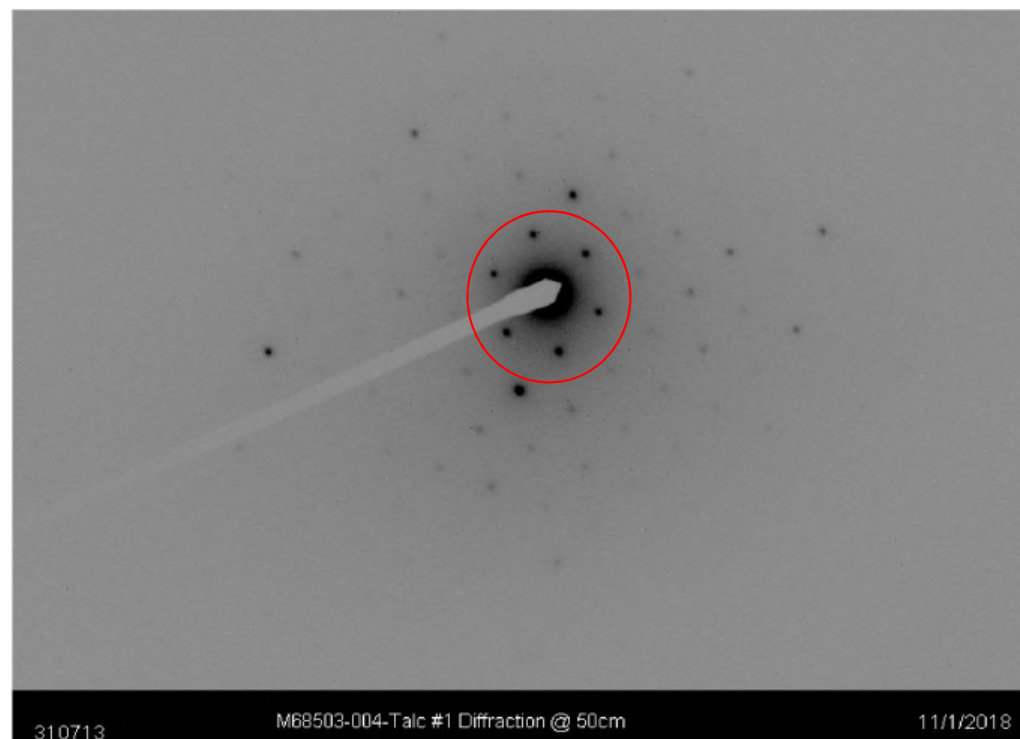
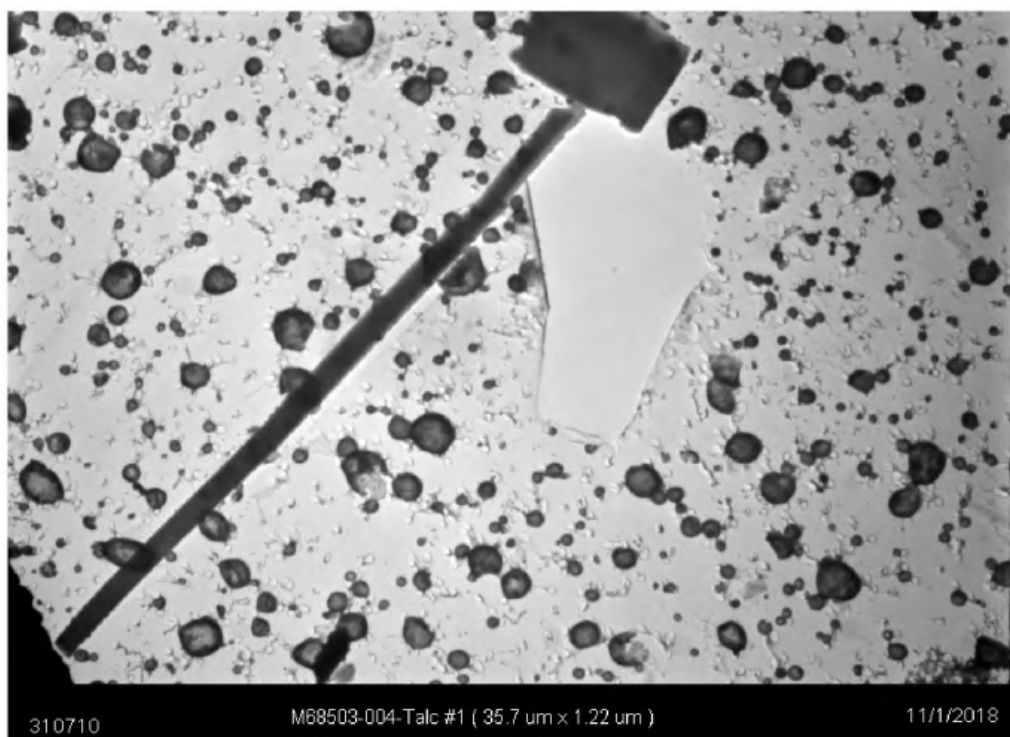


Designation: D5755 – 09 (Reapproved 2014)^{e1}

**Standard Test Method for
Microvacuum Sampling and Indirect Analysis of Dust by
Transmission Electron Microscopy for Asbestos Structure
Number Surface Loading¹**

16.6 Record a **typical electron diffraction pattern** for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

Fibrous Talc in JBP



have similar EDXA spectra. ED of talc produces a pseudo-hexagonal pattern that does not change as the fibre is tilted using the goniometer. Anthophyllite asbestos, on the other hand, produces assorted spots appearing



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00190-191, Tab 9B

INTERNATIONAL
STANDARD

ISO
22262-1

Tab 25, ISO 22262-1, Pg. 64

First edition
2012-07-01

Zone-Axis SAED “seldom” required for lab sample analysis

Analysis of laboratory samples seldom requires zone-axis measurements. However, if a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient



MAS Peer-Reviewed Studies identify asbestos without Zone-Axis

Applied Occupational and Environmental Hygiene
Volume 17(1): 55-62, 2002
Copyright © 2002 Applied Industrial Hygiene
1047-322X/02 \$12.00 + .00

Fiber Release During the Removal of Asbestos-Containing Gaskets: A Work Practice Simulation

William E. Longo, William B. Egeland, Richard L. Hatfield, and Larry R. Newton
Materials Analytical Services, Inc., Suwanee, Georgia

published and unpublished studies previously performed that also used the indirect TEM method.⁽¹⁶⁻¹⁸⁾ The TEM air samples were then analyzed by a modified EPA Level II protocol.⁽¹⁹⁾ Cloth swatches from the work clothing worn by the investigators during the studies were analyzed by the recommended EPA method.⁽²⁰⁾ Surface dust samples were collected from the work

Zone-Axis SAED Not Required

Johnson & Johnson

<i>Johnson & Johnson</i> BABY PRODUCTS COMPANY		T.M. NO. 7024 PAGE 4 of 5
<u>STANDARD TEST METHOD</u>		
SUBJECT: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY <u>TRANSMISSION ELECTRON MICROSCOPY</u>		

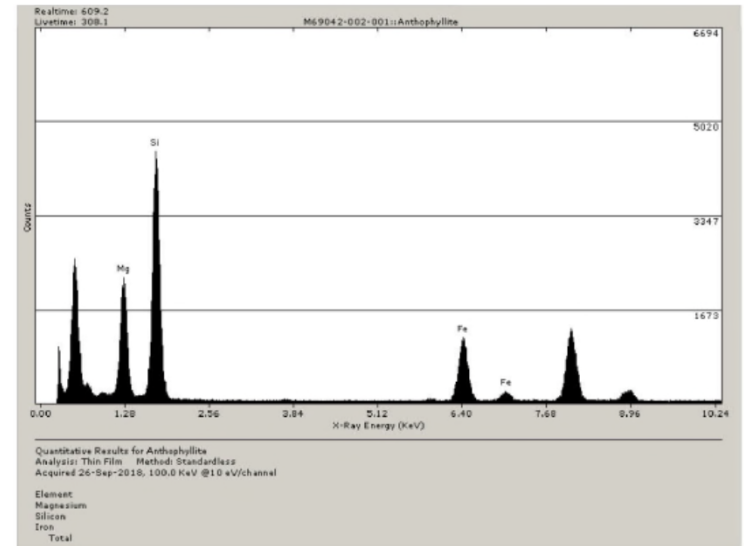
- 13.4. Questionable fibers are examined first by SAED. The chrysotile SAED pattern is unique and diagnostic. Amphibole SAED patterns are variable but usually characteristic. Additional analysis and measurement of amphibole SAED patterns are done if warranted.
- 13.5. Ten percent of chrysotile fibers are checked by EDXRA for further confirmation. If the SAED pattern is not clearly diagnostic, or if it is consistent with an amphibole SAED pattern, then it is examined by EDXRA to confirm the identification or to identify the type of amphibole.

Tab 6, 1989 J&J Test Method TM 7024, Bates: JNJNL61_000043153;
See Also 6A, 1995 J&J Test Method TM 7024, Bates: JNJNL61_000005038

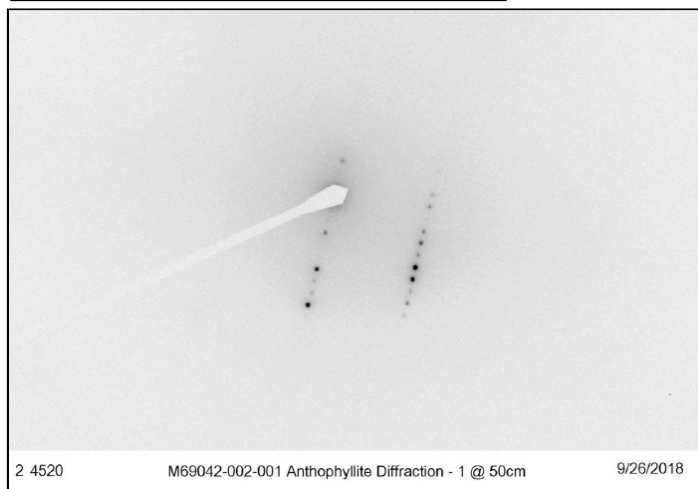
Step 1: Morphology



Step 2: EDXA



Step 3: SAED

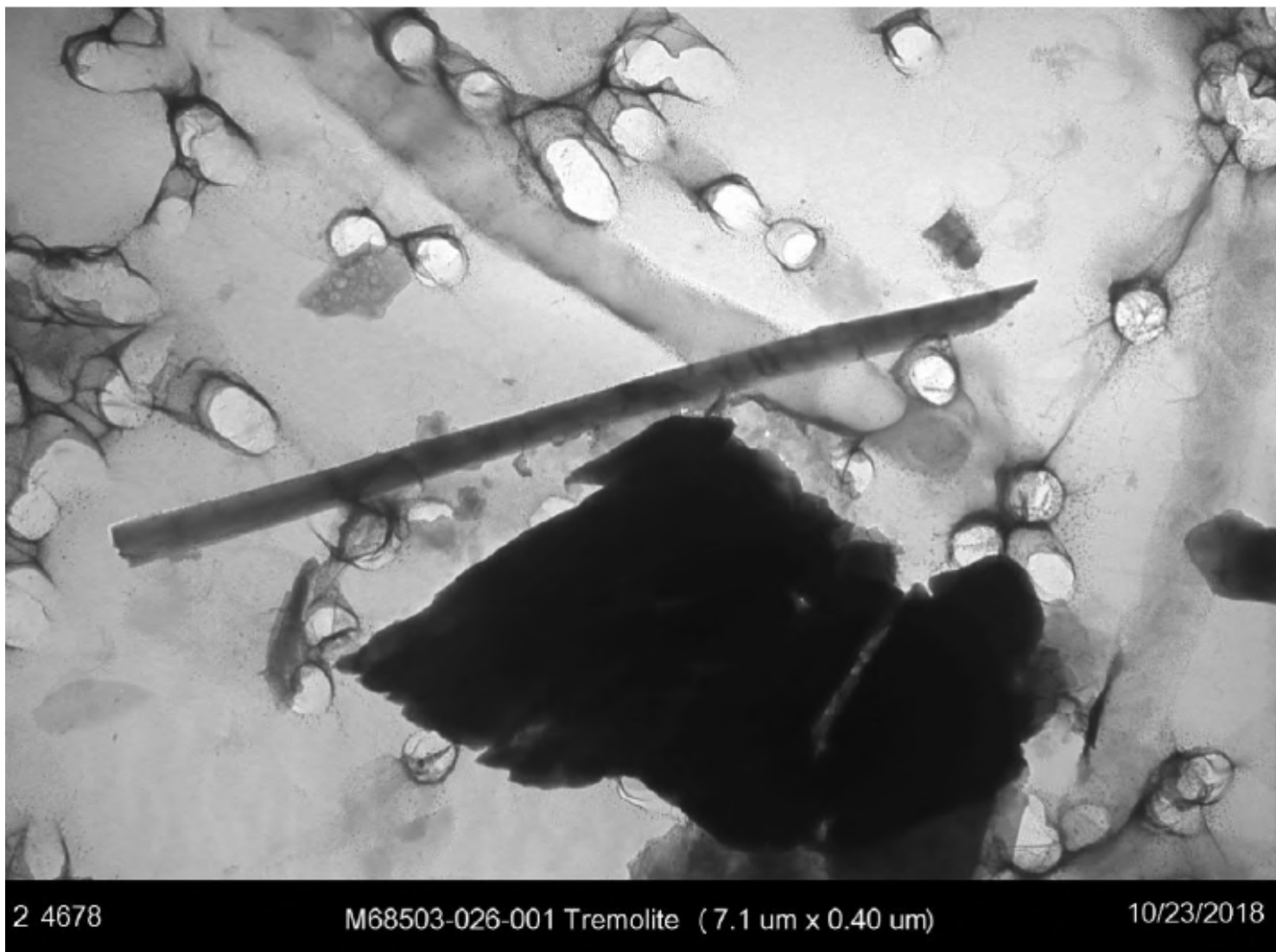


**ANTHOPHYLLITE
= ASBESTOS IN J&J
TALC**

Example 2
Sample M68503-026
1969 Shower to
Shower Sample



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00295-851



Step 1: Morphology

Length: 7.1 μm
Substantially Parallel Sides
Aspect Ratio – 17.8 to 1

MAS 1/15/19 Report Backup
Data Binder,
Bates: Longo-MDL_00326, Tab 9C

2 4678

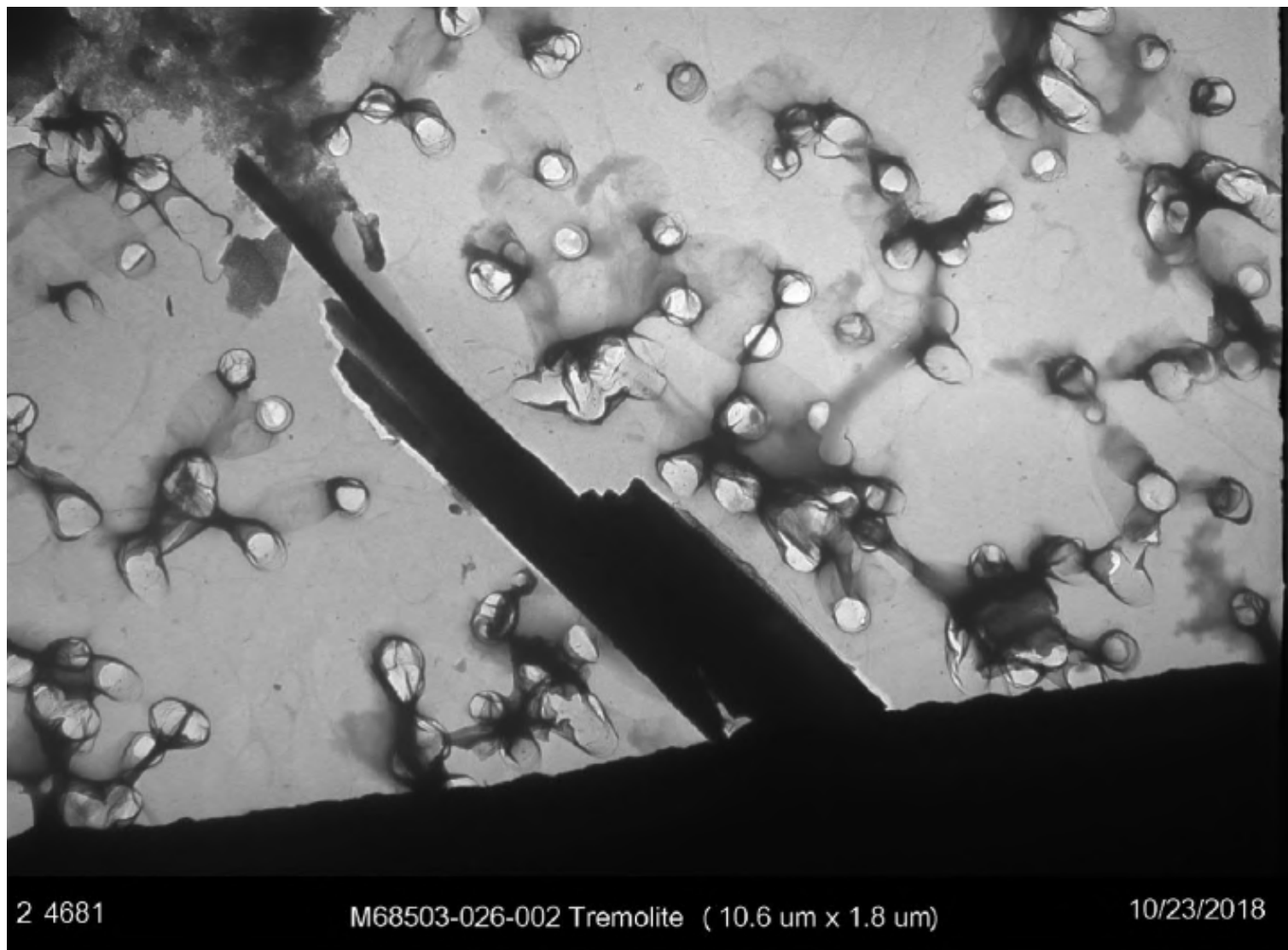
M68503-026-001 Tremolite (7.1 μm x 0.40 μm)

10/23/2018



Step 1: Morphology

Length: 10.6 μm
Substantially Parallel Sides
Aspect Ratio – 5.9 to 1

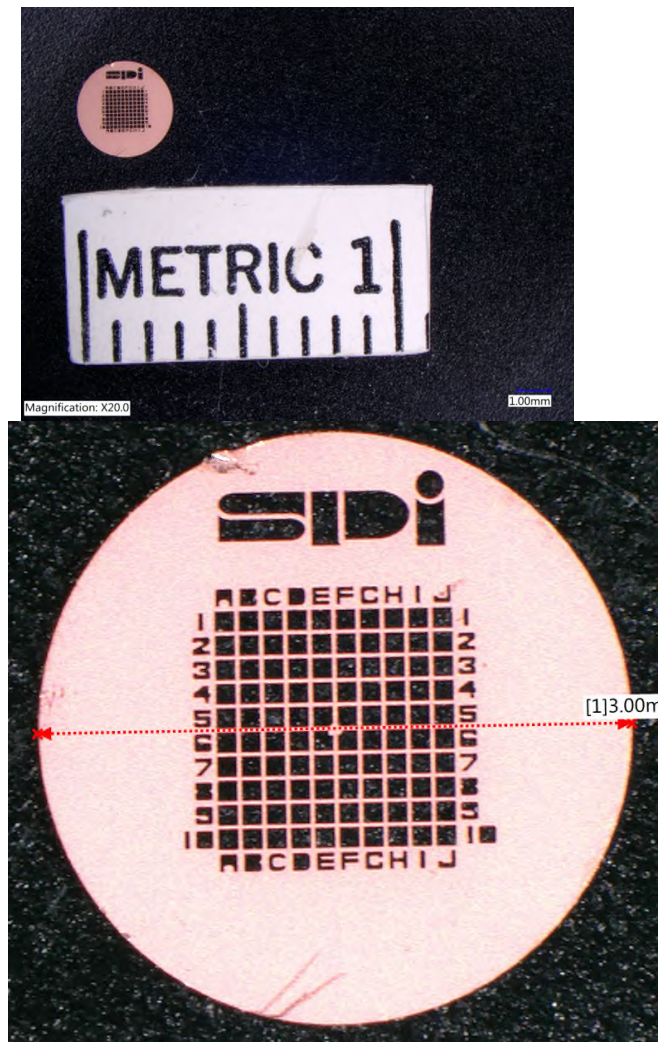


2 4681 M68503-026-002 Tremolite (10.6 μm x 1.8 μm) 10/23/2018

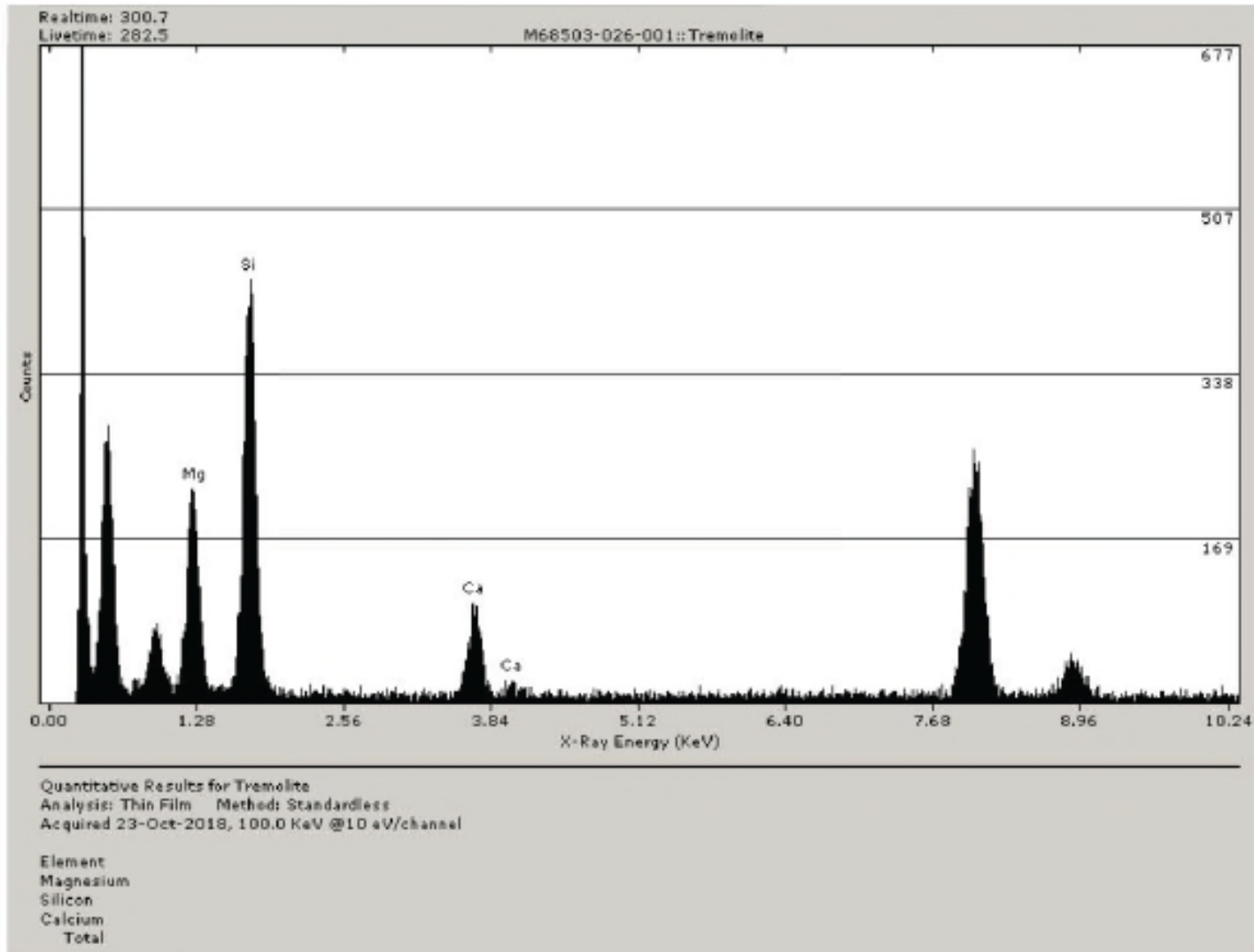
MAS 1/15/19 Report Backup
Data Binder,
Bates: Longo-MDL_00329, Tab
9C

TEM Bulk Talc Structure Count Sheet						
Project/ Sample No.	M68503-026		Grid Box #	8632	No. of Grids Counted	2
Analyst:	Anthony Keeton			Length	Width	G. O. Area
Date of Analysis	10/23/2018 - 10/30/2018		G. O. in microns =	105	105	11025
Initial Weight(g)	0.02109			105	105	11025
Analysis Type	Post Separation Talc Analysis		Grid Acceptance	Yes	Average	11025
Scope No.	Accelerating Voltage	100 KV	Loading%	20%	G.O.s Counted	100
2	Screen Magnification	20 KX	Area Examined mm²			1.103

Str. #	Grid Opening	Structure	Asbestos Type	Length	Width	Ratio	SAED	EDS
NSD	E10-A1							
1	A2	Bundle	Tremolite	7.1	0.40	17.8	X	X
NSD	A3							
NSD	A4							
NSD	A5							
2	A6	Bundle	Tremolite	10.6	1.80	5.9	X	X
3	A7	Fiber	Tremolite	3.1	0.23	13.5	X	X
4		Bundle	Tremolite	7.6	0.80	9.5	X	X
5		Bundle	Tremolite	3.2	0.50	6.4	X	X
NSD	A8							
6	A9	Bundle	Tremolite	7.3	1.20	6.1	X	X
NSD	A10							
NSD	B1							
7	B2	Bundle	Tremolite	7.3	0.70	10.4	X	X
NSD	B3							
NSD	B4							
NSD	B5							
8	B6	Bundle	Tremolite	9.8	1.80	5.4	X	X
9	B7	Bundle	Tremolite	4.3	0.80	5.4	X	X
10	B8	Bundle	Tremolite	7.0	0.80	8.8	X	X
11	B9	Bundle	Tremolite	7.4	1.10	6.7	X	X
NSD	B10							
12	C1	Bundle	Tremolite	13.3	0.70	19.0	X	X
NSD	C2							
13	C3	Bundle	Tremolite	3.7	0.45	8.2	X	X
NSD	C4							
14	C5	Bundle	Tremolite	3.4	0.60	5.7	X	X
15	C6	Bundle	Tremolite	3.2	0.23	13.9	X	X
NSD	C7							



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00321, Tab 9C



Step 2: Chemistry by EDXA

Result: Tremolite Asbestos

MAS 1/15/19 Report Backup Data
Binder, Bates: Longo-
MDL_00324, Tab 9C

INTERNATIONAL
STANDARD

ISO
22262-1

First edition
2012-07-01



9.5 Qualitative analysis by TEM

9.5.5 Tremolite

Classify a fibre as tremolite if:

- a) the Mg, Ca and Fe peaks are comparable in ratio with those of the reference tremolite;
- b) no statistically significant peak from Al is present;
- c) any peak from either Na or K is small.

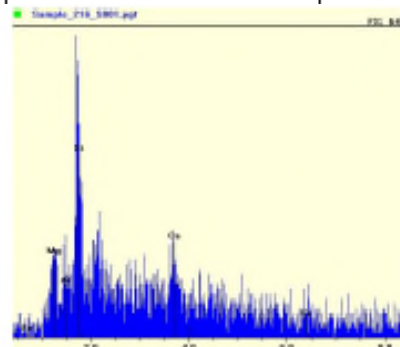
NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

OSHA 2019: “EDX spectrum” (with no SAED) to Identify Tremolite Asbestos

Table 1

Sample Number	Fibers Present in PCM-PLM	Possible amphibole fibers present in PCM-PLM	Fibers Present in SEM	SEM + EDX (chemistry) consistent with regulated minerals	XRD	Regulated asbestos name
761227 Eye shadow	Yes	Yes	Yes	Yes (talc fibers also noted) ¹	ND	Tremolite asbestos

5



6

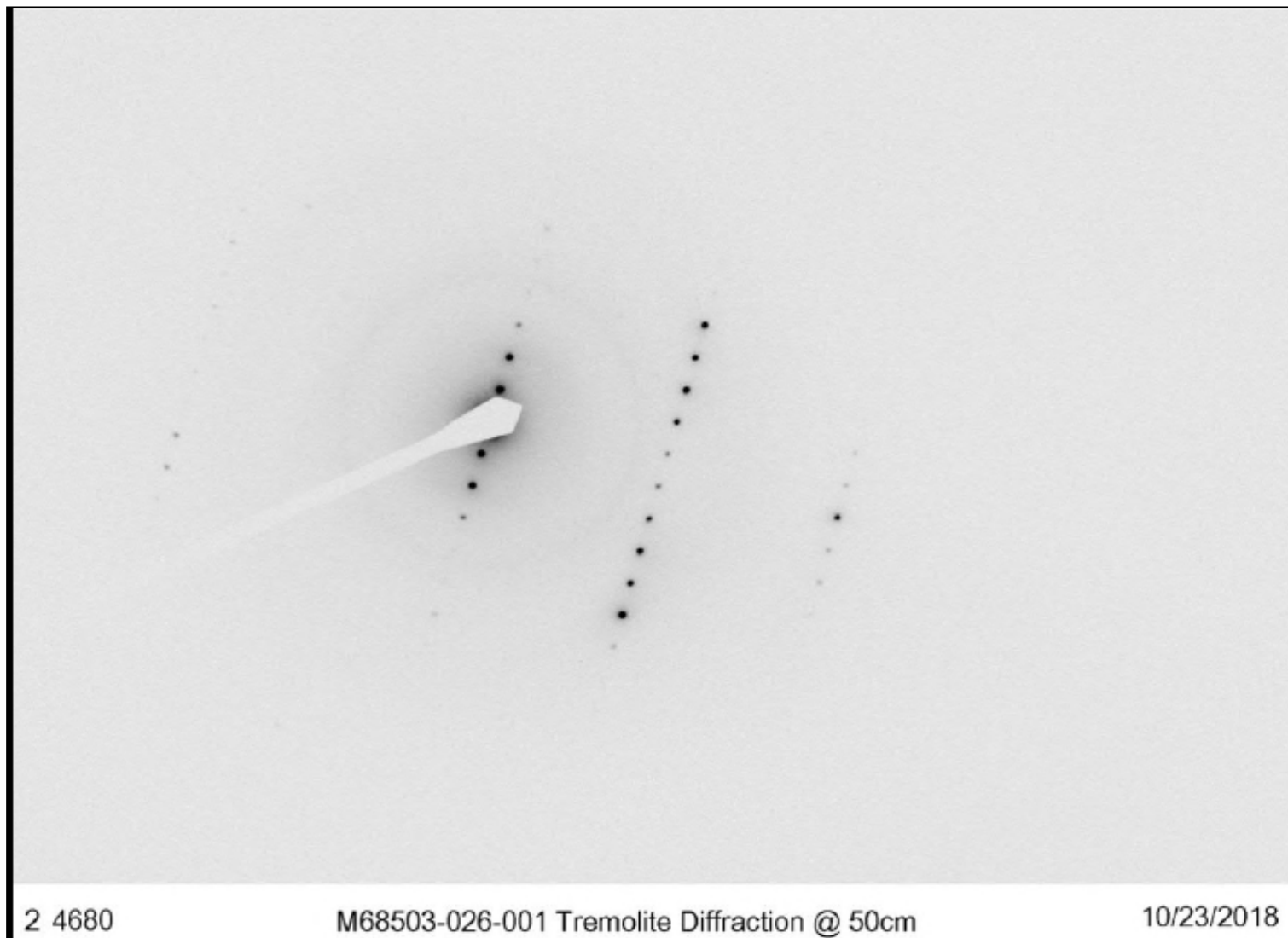
1. Bright Field (PCM) of sample showing a fiber in the center 160X
2. Central Stop Dispersion Stain of the fiber in figure 1. 160X
3. Central Stop Dispersion Stain of a fiber bundle. 160X
4. Crossed polarized light with first order red plate of a fiber in the center 400X
5. SEM of asbestiform tremolite 21 micrometers x 0.7 micrometers
6. EDX spectrum of the fiber in 5, consistent with tremolite.

Tab 26, OSHA’s 2019 Testing of *Claire’s* Talc Products for the FDA, Pg. 3-4



Step 3: Crystal Structure
by SAED

Result: Tremolite Asbestos



2 4680

M68503-026-001 Tremolite Diffraction @ 50cm

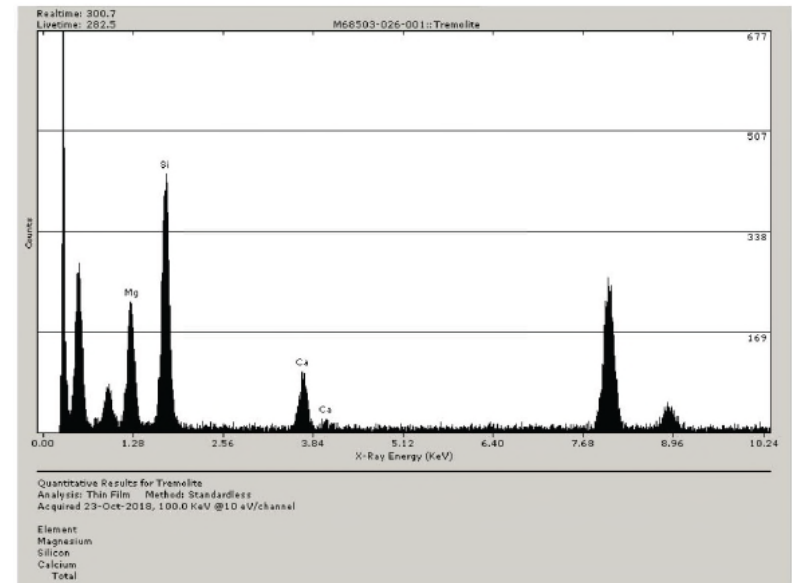
10/23/2018

MAS 1/15/19 Report Backup
Data Binder,
Bates: Longo-MDL_00325,
Tab 9C

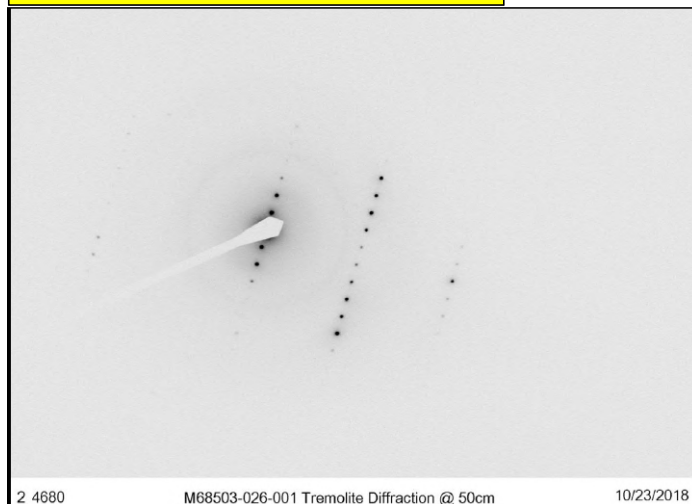
Step 1: Morphology



Step 2: EDXA



Step 3: SAED



**TREMOLITE
=
ASBESTOS IN J&J
TALC**

AHERA Method – How to Identify “Nonasbestos”

#3

(3) Nonasbestos: Incomplete or unobtainable ED patterns, a nonasbestos EDXA, or a nonasbestos morphology.

#2

#1

“Nonasbestos” = when any of the 3 steps are NOT satisfied

All Asbestos identified by MAS in J&J’s Talc satisfied all 3 steps

Has MAS Reliably Identified Asbestos in J&J Talc?

Published TEM Testing Method	Asbestos?
EPA AHERA	YES
ISO 22262-1	YES
ISO 22262-2	YES
ASTM D5755	YES
Johnson & Johnson	Asbestos?
JJ TM 7024	YES

T.M. NO. 7024 B
PAGE 1 of 5

Johnson & Johnson
BABY PRODUCTS COMPANY

5. 'DARD TEST METHOD

SUBJECT: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON MICROSCOPY

Johnson & Johnson

5. 'DARD TEST METHOD

SUBJECT

SUPERS

1. S

T

P

2. E

T

3. I

4. I

5. I

6. I

7. I

8. I

9. I

10. I

11. I

12. I

13. I

14. I

15. I

16. I

17. I

18. I

19. I

20. I

21. I

22. I

23. I

24. I

25. I

26. I

27. I

28. I

PRINCIPLE OF METHOD

#2

#3

The combined techniques of transmission electron microscopy (TEM), selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDXRA) permit the detection of asbestiform minerals based on morphological characteristics, followed by a definitive mineralogical identification of each fiber.

INTERFERENCES

Interferences are caused by fibrous particles which must be distinguished from positively identifiable asbestos, and by large particles or particle aggregates which may obscure fibers. Positively identified non-asbestos fibers include rolled talc, ribbon talc, antigorite, silica fibers and iron oxide fibers. Organic

#1

TEM ANALYSIS

#1

13.1 **Definition of fiber:** An elongated particle with parallel sides and an aspect ratio >3:1. The definition employed may vary with the needs of

10^{-14} g for chrysotile and 1.5×10^{-14} g for amphibole.

This method is capable of detecting a single fiber as small as 1 micrometer (μm)

MAS used the more restrictive AHERA 5:1 aspect ratio

J&J-0007919

JNJNL61_000043150

Protected Document—Subject to Protective Order

Tab 6, 1989 J&J Test Method TM 7024, Bates: JNJNL61_000043150-43151
See Also, Tab 6A, 1995 J&J Test Method TM 7024, Bates: JNJNL61_000005035, 5038

ISO 22262-2 – Single Asbestos Fiber by TEM is Sufficient to Determine Presence of Asbestos

Table 1 — Summary of requirements for quantification of asbestos in bulk samples

Type of material	Regulatory control limit			
	"Any asbestos"	Mass fraction > 0,1 %	Mass fraction > 0,5 %	Mass fraction > 1 %
Commercially manu- factured product	If any commercial asbestos variety is detected, no further quantification is required		If asbestos is detected at an estimated mass fraction of < 5 %, more precise quantifica- tion is required to establish the regulatory status of the material	
Other materials	If any variety of asbestos is detected, no further quanti- fication is required	If asbestos is detected at an estimated mass fraction of < 5 %, more precise quantification is required to establish the regulatory status of the material		

7 Limit of quantification

The limit of quantification using this part of ISO 22262 is defined as the detection and identification of one fibre or fibre bundle in the amount of sample examined. The limit of quantification that can be

INTERNATIONAL
STANDARD

ISO
22262-2

Tab 18, ISO 22262-2 Pg. 7

T.M. NO. 7024 B
PAGE 1 of 5

Johnson & Johnson
BABY PRODUCTS COMPANY

5. STANDARD TEST METHOD

SUBJECT: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON MICROSCOPY

Johnson & Johnson

T.M. NO. 7024 B
PAGE 1 of 5

Johnson & Johnson
BABY PRODUCTS COMPANY

5. STANDARD TEST METHOD

SUBJECT: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON MICROSCOPY

SUPERSEDES: ADL 1305 DATE: 3/8/89 AUTHORIZATION NO.: BCR 011362

1. SCOPE & PURPOSE

This method is applicable to the identification and quantitation of small (typically 1-20 micrometer) asbestiform minerals in powdered talc. Samples may be previously screened with light microscopy or x-ray diffraction techniques.

2. PRINCIPLE OF METHOD

The combined techniques of transmission electron microscopy (TEM), selected area

3. **6. LIMIT OF QUANTIFIABLE DETECTION**

The detection of five or more asbestiform minerals of one variety in an analysis constitutes a quantifiable level of detection. When no asbestiform minerals are

4. INSTRUMENTAL CONDITIONS

The talc specimen grids are examined in the TEM at an accelerating voltage of 120 kv and at magnification of 20,000X and 5,000X.

5. SENSITIVITY

This method is capable of detecting a single fiber as small as 1 micrometer (μm) long by 0.075 μm wide in the entire TEM field, which results in a theoretical detection limit of 10^{-3} weight percent. Such fibers usually can be identified readily by SAED and EDXRA. The mass of a fiber with the above dimensions is 1.1×10^{-14} g for chrysotile and 1.5×10^{-14} g for amphibole.

J&J-0007919
JNJNL61_000043150

Protected Document-Subject to Protective Order

J&J Method Reports Results as “non-quantifiable” if less than 5 fibers of same asbestos type not detected. Results in “false negatives” and unreliable test results

Tab 6, 1989 J&J Test Method TM 7024, Bates: JNJNL61_000043150-43151
See Also, Tab 6A, 1995 J&J Test Method TM 7024, Bates: JNJNL61_000005035

There is no test for “High Tensile Strength” and “Flexibility” in the published methods

- Would exclude noncommercial amphibole asbestos which are known to have low tensile strength and poor flexibility (i.e. not of commercial value)
- Often included in general definition of “asbestos” to identify commercial attributes

Commercial asbestos deposits sometimes consist of magnesium-rich actinolite and tremolite. Although of low tensile strength and poor spinnability, high resistance to acids may make the fibres suitable for acid filtration. Physical and optical characteristics are listed in Table 1.

Table 1

PHYSICAL PROPERTIES OF ASBESTOS (AFTER BADOLLET 1951)

	Chrysotile	Amosite	Anthophyllite	Crocidolite	Tremolite	Actinolite
Specific heat B.t.u. per lb. per °F..	0.266	0.193	0.210	0.201	0.212	0.217
Tensile strength, lb. per sq. in. ...	80,000-100,000	16,000-90,000	4,000 or less	100,000-300,000	1,000-8,000	1,000 or less
Flexibility	nickel, lime High	Good	Poor	Good	Poor	Poor
Resistance to heat	Good,	Good,	Very good	Poor, fuses	Fair to good	...

Tab 51, 1971 Report entitled “Asbestos in Ontario”, Pg. 9, 11

There is no test for “High Tensile Strength” and “Flexibility” in the published methods

- Would exclude noncommercial amphibole asbestos which are known to have low tensile strength and poor flexibility i.e not of commercial value
- Often included in general definition of “asbestos” to identify commercial attributes

MINERALOGY OF ASBESTOS

The term "asbestos" is applied to minerals of the serpentine and amphibole groups with the common property of naturally occurring in a fibrous habit. The fibres must be strong, flexible and sufficiently heat resistant to be of commercial value. The longer fibres of good quality, particularly those of chrysotile, a fibrous form of serpentine, can be woven into textiles. Many

AHERA Method – Asbestos Structures can be Fibers, Bundles, Clusters, or Matrices

F. TEM Method

9. *Recording Rules.*

a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 μm is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any



ASTM D5755 – Asbestos Structures can be Fibers, Bundles, Clusters of Matrices



Designation: D5755 – 09 (Reapproved 2014)^{e1}

Standard Test Method for
Microvacuum Sampling and Indirect Analysis of Dust by
Transmission Electron Microscopy for Asbestos Structure
Number Surface Loading¹

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the USEPA Asbestos-Containing Materials in Schools document (4). Record, for each AHERA structure identified, the length and width measurements.

3.2.6 *fiber*—a structure having a minimum length of 0.5 μm , an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

**MAS TEM Coefficient of Variation for Tremolite and Anthophyllite in Talc
A Quality Control Study**

9-6-18



The purpose of this Quality Control study was to determine the MAS TEM analysis coefficient of variation (CV or relative standard deviation RSD) after spiking cosmetic grade talc powder with tremolite and anthophyllite asbestos standard reference material (SRM).

TABLE 1 Total Structures and Structures per gram of Tremolite and Anthophyllite in Talc Powder Samples

Sample	Component	Str/g			
		Analyst 1	Analyst 2	Analyst 3	Analyst 4
0.3%	Tremolite	3.20E+05	3.55E+05	3.20E+05	3.55E+05
0.3%	Anthophyllite	4.90E+05	5.39E+05	4.90E+05	5.39E+05

TABLE 2 Average, SD and CV for the TEM Analysis of Tremolite and Anthophyllite in Talc Powder Samples

Sample	Component	Str/g		
		Mean	STD	CV (%)
0.3%	Tremolite	3.38E+05	2.0E+04	5.99
0.3%	Anthophyllite	5.14E+05	2.8E+04	5.50

Tab 52,
MAS Coefficient
of Variation Report,
dated 9.6.18

J&J's Chart from C of V Report Regarding Tremolite Asbestos Bundles vs. Fibers

Grid Opening	(Analyst 1) Structure	(Analyst 2) Structure	(Analyst 3) Structure	(Analyst 4) Structure
A8-E2	Bundle	Fiber	Fiber	Bundle
A8-E4	Fiber	Fiber	Fiber	Fiber
A8-E5	Bundle	Bundle	Bundle	Fiber
A8-E7	Fiber	Bundle	Fiber	Fiber
A8-E8	Bundle	Fiber	Bundle	Fiber
A8-E9	Bundle	Bundle	Bundle	Fiber
A8-F2	Bundle	Bundle	Fiber	Bundle
A8-G1	Bundle	Bundle	Bundle	Fiber
A8-G4				Fiber
A8-G5		Bundle		
A8-G6	Bundle	Fiber	Bundle	Bundle

Tab 53, Excerpt from J&J Motion to Exclude Plaintiff's Experts' Asbestos-Related Opinions, Pg. 47, Dated 5/7/19

FIBERS v BUNDLES COMPARISON

0.3% TREMOLITE

25 GOs

Project/ Sample No.	M65947-001
------------------------	------------

Keeton

Grid Opening	Structure
A8-E1	
A8-E10	
A8-E2	Bundle
A8-E3	
A8-E4	Fiber
A8-E5	Bundle
A8-E6	
A8-E7	Fiber
A8-E8	Bundle
A8-E9	Bundle
A8-F10	
A8-F2	Bundle
A8-F3	
A8-F4	
A8-F5	
A8-G1	Bundle
A8-G10	
A8-G2	
A8-G3	
A8-G4	
A8-G5	
A8-G6	Bundle
A8-G7	
A8-G8	
A8-G9	

Motamedi

Grid Opening	Structure
A8-E1	
A8-E10	
A8-E2	Fiber
A8-E3	
A8-E4	Fiber
A8-E5	Bundle
A8-E6	
A8-E7	Bundle
A8-E8	Fiber
A8-E9	Bundle
A8-F1	
A8-F10	
A8-F2	Bundle
A8-F3	
A8-F4	
A8-F5	
A8-G1	Bundle
A8-G10	
A8-G2	
A8-G3	
A8-G4	
A8-G5	Bundle
A8-G6	Fiber
A8-G7	
A8-G8	
A8-G9	

Carillo

Grid Opening	Structure
A8-E1	
A8-E10	
A8-E2	Fiber
A8-E3	
A8-E4	Fiber
A8-E5	Bundle
A8-E6	
A8-E7	Fiber
A8-E8	Bundle
A8-E9	Bundle
A8-F1	
A8-F10	
A8-F2	Fiber
A8-F3	
A8-F4	
A8-F5	
A8-G1	Bundle
A8-G10	
A8-G2	
A8-G3	
A8-G4	
A8-G5	
A8-G6	Bundle
A8-G7	
A8-G8	
A8-G9	

Callan

Grid Opening	Structure
A8-E1	
A8-E10	
A8-E2	Bundle
A8-E3	
A8-E4	Fiber
A8-E5	Fiber
A8-E6	
A8-E7	Fiber
A8-E8	Fiber
A8-E9	Fiber
A8-F1	
A8-F10	
A8-F2	Bundle
A8-F3	
A8-F4	
A8-F5	
A8-G1	Fiber
A8-G10	
A8-G2	
A8-G3	
A8-G4	Fiber
A8-G5	
A8-G6	Bundle
A8-G7	
A8-G8	
A8-G9	

Grid Opening (Ordered)	% Agreement
A8-E1	
A8-E10	
A8-E2	50
A8-E3	
A8-E4	100
A8-E5	75
A8-E6	
A8-E7	75
A8-E8	50
A8-E9	75
A8-F1	
A8-F10	
A8-F2	75
A8-F3	
A8-F4	
A8-F5	
A8-G1	75
A8-G10	
A8-G2	
A8-G3	
A8-G4	NA
A8-G5	NA
A8-G6	75
A8-G7	
A8-G8	
A8-G9	
AVE	72.2

Tab 52A, Chart
summarizing
results from
Tab 52, Pg. 1

FIBERS v BUNDLES COMPARISON 0.3% ANTHOPHYLLITE 25 GOs

Project/ Sample No.	M65947-002
------------------------	------------

Keeton		Motamedi		Carillo		Callan		Grid Opening (Ordered)	% Agreement
Grid Opening	Structure	Grid Opening	Structure	Grid Opening	Structure	Grid Opening	Structure		
A4-A1	Fiber	A4-A1	Bundle	A4-A1	Fiber	A4-A1	Fiber	A4-A1	75
A4-A2		A4-A1	Bundle	A4-A1	Bundle	A4-A1	Bundle	A4-A1	100
A4-A10		A4-A10		A4-A10		A4-A10		A4-A10	
A4-A2		A4-A2		A4-A2		A4-A2		A4-A2	
A4-A3		A4-A3		A4-A3		A4-A3		A4-A3	
A4-A4	Bundle	A4-A4	Fiber	A4-A4	Bundle	A4-A4	Bundle	A4-A4	75
A4-A5	Bundle	A4-A5	Bundle	A4-A5	Bundle	A4-A5	Bundle	A4-A5	100
A4-A6	Bundle	A4-A6	Bundle	A4-A6	Bundle	A4-A6	Bundle	A4-A6	100
A4-A7	Bundle	A4-A7	Bundle	A4-A7	Bundle	A4-A7	Bundle	A4-A7	100
A4-A8		A4-A8		A4-A8		A4-A8		A4-A8	
A4-A9	Bundle	A4-A9	Bundle	A4-A9	Bundle	A4-A9	Bundle	A4-A9	100
A4-D10	Bundle	A4-D10	Bundle	A4-D10	Bundle	A4-D10	Bundle	A4-D10	100
A4-D6		A4-D6		A4-D6		A4-D6		A4-D6	
A4-D7	Bundle	A4-D7	Bundle	A4-D7	Fiber	A4-D7	Bundle	A4-D7	75
A4-D8	Bundle	A4-D8	Bundle	A4-D8	Bundle	A4-D8	Bundle	A4-D8	100
A4-D8	Bundle	A4-D8	Bundle	A4-D8	Fiber	A4-D8	Bundle	A4-D8	75
A4-D9	Fiber	A4-D9	Fiber	A4-D9	Fiber	A4-D9	Bundle	A4-D9	75
A4-D9	Fiber	A4-D9	Bundle	A4-D9	Fiber	A4-D9	Fiber	A4-D9	75
A4-E1		A4-E1		A4-E1		A4-E1		A4-E1	
A4-E10	Bundle	A4-E10	Fiber	A4-E10	Bundle	A4-E10	Fiber	A4-E10	50
A4-E10	Fiber	A4-E10	Bundle	A4-E10	Fiber	A4-E10	Bundle	A4-E10	50
A4-E2	Fiber	A4-E2	Fiber	A4-E2	Fiber	A4-E2	Fiber	A4-E2	100
A4-E3	Fiber	A4-E3	Fiber	A4-E3	Fiber	A4-E3	Fiber	A4-E3	100
A4-E4		A4-E4		A4-E4		A4-E4		A4-E4	
A4-E5		A4-E5		A4-E5		A4-E5		A4-E5	
A4-E6	Fiber	A4-E6	Fiber	A4-E6	Fiber	A4-E6	Fiber	A4-E6	100
A4-E7	Bundle	A4-E7	Bundle	A4-E7	Bundle	A4-E7	Bundle	A4-E7	100
A4-E8		A4-E8		A4-E8		A4-E8		A4-E8	
A4-E9	Fiber	A4-E9	Bundle	A4-E9	Fiber	A4-E9	Bundle	A4-E9	50
A4-E9	Bundle	A4-E9	Fiber	A4-E9	Bundle	A4-E9	Bundle	A4-E9	75
A4-E9	Bundle	A4-E9	Bundle			A4-E9	Fiber	A4-E9	67
AVE									83.7

Tab 52A, Chart
summarizing results
from Tab 52, Pg. 2

J3 Resources Verification of MAS 22262-2 Heavy Liquid TEM Testing of J&J Products



Summary of Results

All 22 particles were readily located in the grid openings as originally reported. Additionally, the dimensions measured during the VAA approximately matched the measurements originally reported. While there was 100% agreement the 22 asbestos structures reported were anthophyllite and tremolite, I judged 2 of the 22 structures did not meet the strict definition of a regulated asbestos fiber (parallel sides). I was able to complete my VAA. Overall, this VAA yielded a >90% validation rate. The table



J3 Resources
Verification
of MAS
22262-2
Heavy Liquid
TEM Testing
of J&J
Products



**Summary of Verified Analysis of 22 Asbestos Structures Detected in Six
Historical Johnson's Baby Powder Originally Analyzed by MAS, LLC**

SAMPLE #	STR #	GO	DIMENSION (µM)	STR. TYPE	ID	VERIFIED
M69042-001	1	B8	14 x 0.4	B/Grid Bar	Anth	Yes
	2	D10	2 x 0.4	B	Anth	Yes
	3	A2	15.5 x 2.0	B	Anth	Yes
	4	C6	10 x 0.25	F	Anth	Yes
	5	C10	22 x 2.5	Cleav Frag/Trans	Anth/Talc	No
M69042-002	1	B8	35 x 1.5	B	Anth	Yes
	2	B8	12 x 1.0	B	Anth	Yes
	3	E1	6 x 1.0	F	Anth	Yes
	4	E9	5.5 x 0.6	B	Anth	Yes
	5	H7	32 x 0.9	B	Anth	Yes
	6	C1	10 x 1.1	B	Anth	Yes
	7	G3	10.5 x 1.0	B	Anth	Yes
M69042-003	1	A8	4.5 x 0.4	Cleav Frag	Trem	No
	2	F3	3.2 x 0.4	B	Anth	Yes
M69042-004	1	A8	14 x 0.4	B	Anth	Yes
	2	F1	4 x 0.35	B	Anth	Yes
	3	E3	13 x 0.5	B	Anth	Yes
M69042-008	1	C2	4 x 0.5	B	Anth	Yes
	2	B1	8 x 1.5	B	Anth	Yes
	3	C6	5 x 0.5	B	Anth	Yes
M69042-010	1	F5	9 x 1.5	B	Anth	Yes
	2	A9	8.5 x 0.5	B	Anth	Yes



J3 verified as asbestos 20 out of the 22 asbestos structures identified by MAS. (91%)

Out of the 20 Agreed-Upon Asbestos Structures:

- MAS and J3 agreed on the asbestos structure type for 16 out of 20. (80%)
- J3 identified 18 out of 20 as bundles. (90%)
- MAS identified 16 out of 20 as bundles. (80%)



Summary of Verified Analysis of 22 Asbestos Structures Detected in Six Historical Johnson's Baby Powder Originally Analyzed by MAS, LLC							MAS Str. Type
SAMPLE #	STR #	GO	DIMENSION (µM)	STR. TYPE	ID	VERIFIED	
M69042-001	1	B8	14 x 0.4	B/Grid Bar	Anth	Yes	F
	2	D10	2 x 0.4	B	Anth	Yes	F
	3	A2	15.5 x 2.0	B	Anth	Yes	B
	4	C6	10 x 0.25	F	Anth	Yes	F
	5	C10	22 x 2.5	Cleav Frag/Trans	Anth/Talc	No	B
M69042-002	1	B8	35 x 1.5	B	Anth	Yes	B
	2	B8	12 x 1.0	B	Anth	Yes	B
	3	E1	6 x 1.0	F	Anth	Yes	B
	4	E9	5.5 x 0.6	B	Anth	Yes	B
	5	H7	32 x 0.9	B	Anth	Yes	B
	6	C1	10 x 1.1	B	Anth	Yes	B
	7	G3	10.5 x 1.0	B	Anth	Yes	B
M69042-003	1	A8	4.5 x 0.4	Cleav Frag	Trem	No	B
	2	F3	3.2 x 0.4	B	Anth	Yes	B
M69042-004	1	A8	14 x 0.4	B	Anth	Yes	F
	2	F1	4 x 0.35	B	Anth	Yes	B
	3	E3	13 x 0.5	B	Anth	Yes	B
M69042-008	1	C2	4 x 0.5	B	Anth	Yes	B
	2	B1	8 x 1.5	B	Anth	Yes	B
	3	C6	5 x 0.5	B	Anth	Yes	B
M69042-010	1	F5	9 x 1.5	B	Anth	Yes	B
	2	A9	8.5 x 0.5	B	Anth	Yes	B

J3 Resources, Inc. 22262-2 Heavy Liquid TEM Testing of J&J Talc Products

ATEM of Historical J&J Vermont Talc Shower to Shower Talcum Powder

On July 18, 2018 Lee Poye of J³ Resources, Inc. issued a report (to Joe Satterley of the Kazan Law Firm) of his analysis of 15 historical J&J Vermont talc Shower to Shower talcum powder samples that were split by J&J from their historical Shower to Shower (STS) containers that ranged in date from 1978 to 1986.¹⁶

Of the 15 STS containers (16 samples) analyzed by Lee Poye using the ISO 22262-2 heavy liquid TEM method, 11 of the 16 samples were positive for anthophyllite asbestos (solid solution



Tab 9, MAS 1/15/19 Report, Pg. 14



Tab 29A, J3 Resources 7/13/18 Report

MAS Verification of J3 Resources, Inc. 22262-2 Heavy Liquid TEM Testing of J&J Products

Laboratory Control Number	J&J Sample Identification Number	STS Container Year	Mass Fraction Percent Wt.	Anthophyllite Asbestos (f/b) Concentration per g
20180070-07D	2014.001.0397	1978	7.3×10^{-4}	82,370
20180061-37D	STS001	1982	3.0×10^{-5}	9,257
20180061-38D	STS002	1980	3.0×10^{-3}	53,416
20180061-45D	STS009	1982	1.9×10^{-3}	9,000
20180061-52D	STS016	1980 - 1981	4.0×10^{-3}	70,126
20180061-63D	STS027	1980	3.5×10^{-5}	7,419
20180061-65D	STS029	1980 - 1981	9.2×10^{-3}	95,321
20180061-10D	STS044	1980 - 1981	2.6×10^{-5}	12,209
20180061-15D	STS049	1978	1.3×10^{-3}	60,507
20180061-31F	STS065	1986	2.9×10^{-3}	21,964
20180061-31G	STS065	1986	5.2×10^{-4}	29,715



MAS was able to verify nine of the 11 ATEM positive historical STS talcum powder samples reported by J³. The nine positive MAS verified STS ATEM samples, two non-verified STS positive

Tab 29A, J3 Resources 7/13/18 Report

Tab 9, MAS 1/15/19 Report, Pg. 15

MAS TEM Testing Results



ATEM ISO 22262-2 Method

The ISO 22262-2 ATEM heavy liquid separation method showed that out of the 70 historical JBP/STS containers and Imerys' railroad cars samples, 42 (60 %) contained regulated asbestos fibers and bundles. Two types of asbestos amphiboles were detected in these samples, they were either the tremolite asbestos solid solution series and or the anthophyllite solid solution series asbestos. Only the iron-rich anthophyllite asbestos was detected in the ATEM.

The amphibole asbestos structures per gram of talc ranged from below our analytical sensitivity/detection limit of approximately 3,000 - 9,400 fibers/bundles per gram to an amphibole asbestos concentration that ranged from 4,400 - 268,000 fibers-bundles/gram of talc. Also, for the positive ATEM samples the results were also expressed as a weight percent.

amphibole asbestos detected in 42 of 71 (60%)

How many asbestos structures per gram?

Example 1
Sample M69042-02
1978 JBP Sample



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00853-909

Org. Sample Wt.	Sample Wt. Post HL Separation	
0.02000	0.02000	g
Percent of Orig. Post Separation	100	(%)
Wt. Of Sample Analyzed	0.00010965	g
Filter size	201.1	mm ²
Number of Structures Counted	7	Str.
Structures per Gram of Sample	6.38E+04	Str./g

63,800 asbestos
structures per gram
of JBP

16,278,570 asbestos structures in a 9 oz. bottle

- 63,800 structures per gram
- 28.35 grams per ounce
- 1,808,730 structures per ounce
- 9 ounce bottle
- 16,278,570 asbestos structures in a 1978 (Vermont talc) nine ounce bottle of Johnson's Baby Powder



PLM Analytical Procedure



ISO-22262-1 PLM (MAS)

Approximately 60 to 100 milligrams each of the 56 talcum powder samples were analyzed by the ISO 22262-1 PLM method. Three mounts of the talcum powder sample are placed on two glass slides, a drop of the 1.605 refractive index fluid was placed onto each of the three talcum powder mounts, stirred with the point of a scalpel blade, and then covered with an 18 x 18 mm glass cover slip. The entire area of the three coverslip mounts were examined (972 mm²). Positive identification of amphibole asbestos was done by morphology, refractive indices, elongation, angle of extinction, and birefringence. For positive samples, a visual estimation of the quantity of asbestos observed was based on eye calibration through review of lab generated weight percent standards. Visual calibration was augmented by the use of area percent charts.

ISO 22262-1 – 1.605 Refractive Index Fluid is the Right Fluid to Use for Non-commercial Amphibole Asbestos

7.2.3.5 Preparation of samples for PLM examination

A tentative identification based on the stereomicroscope evaluation is used to select the most appropriate RI mounting liquid. Fibres selected shall be dry and relatively free from other particulate matter. Representative fibres or fibre bundles are chosen and are placed on a clean microscope slide into a drop of RI liquid, and a clean cover glass is lowered gently onto the slide, avoiding trapping of air bubbles. The RI of the liquid selected should be 1,550 for suspected chrysotile, 1,680 for suspected amosite, 1,700 for suspected crocidolite, 1,605 for suspected tremolite or anthophyllite, and 1,630 for suspected actinolite or richterite/winchite.

INTERNATIONAL
STANDARD

ISO
22262-1

First edition
2012-07-01



1.605 Refractive Index Fluid is the Right Fluid to Use for Non-commercial Amphibole Asbestos

U.S. Department of Labor

**Occupational Safety and Health
Salt Lake Technical Center
Sandy, Utah 84070**



**Report of Evaluation of Cosmetics and Cosmetic Talc for FDA
Daniel T Crane
23 February 2019**

The samples were examined by phase contrast with polarized light illumination (PCM-PLM). The test used is standard at the Salt Lake Technical Center for screening talc samples for amphibole asbestos.^{1,2} Samples are mounted in index of refraction liquid $n = 1.605$ and analyzed using central stop dispersion staining (CSDS). Generally, if any fibers are present which have



Example 1

Sample M69042-02

1978 JBP Sample



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00853-909



INTERNATIONAL STANDARD

ISO
22262-1

First edition
2012-07-01



MAS, LLC PLM ANALYSIS

Proj#-Spl# M69042 - 002BL Analyst Paul Hess Date 10/15/2018
ClientName LEVY & KONIGSBERG ClientSpl 20180056-06D
Location _____
Type_Mat Johnson & Johnson Talcum Powder
Gross White debris on slide % of Sample 100
Visual _____

OPTICAL DATA FOR ASBESTOS IDENTIFICATION

Morphology	straight	straight	
Pleochroism	none	none	
Refract Index	1.630/1.615	1.630/1.615	
Sign^	positive	positive	
Extinction	oblique	parallel	
Birefringence	moderate	moderate	
Melt	no	no	
Fiber Name	Actinolite/Tremolite	Anthophyllite	

ASBESTOS MINERALS

EST. VOL. %

Chrysotile.....	_____
Amosite.....	_____
Crocidolite.....	_____
Tremolite/Actinolite.....	<0.1
Anthophyllite.....	<0.1

Comments Actinolite/Tremolite and Anthophyllite asbestos observed. X=Materials Detected.

ISO 22262-1:

7.2.3.6 Identification of asbestos by PLM and dispersion staining

Identification of a **single asbestos fibre** requires the observation of the following properties in the stated observation modes:

- a) **morphology** — observed in all illumination conditions;
- b) **colour and pleochroism** — observed in plane polarized light;
- c) **birefringence** — observed with crossed polars;
- d) **extinction characteristics** — observed with crossed polars;

NOTE The extinction characteristics can also be observed with crossed polars and a 530 nm retardation plate inserted. Under these conditions, when the interference colour of the fibre matches the background colour, the fibre is at the extinction position.

- e) **sign of elongation** — observed with crossed polars and a 530 nm retardation plate inserted;
- f) **refractive indices** — assessed using a dispersion staining objective with polarizer only inserted.

MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_00854, Tab 9F

Tab 25, ISO 22262-1 Pg. 21

ISO 22262-1 – Morphology by PLM

7.2.3.7.1 Morphology

A detailed description for the morphology that is characteristic of asbestos is as follows. This morphology is characteristic of the larger fibres seen in stereomicroscope examinations and of fibres selected from laboratory samples for PLM identification of fibre type.

In the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- a) the presence of fibre aspect ratios in the range of 20:1 or higher for fibres longer than 5 µm;
- b) the capability of longitudinal splitting into very thin fibrils, generally less than 0,5 µm in width;
- c) in addition, observation of any of the following characteristics for the fibre type under consideration provides additional confirmation that the fibres are asbestiform:
 - 1) parallel fibres occurring in bundles,
 - 2) fibre bundles displaying splayed ends,
 - 3) fibres in the form of thin needles,
 - 4) matted masses of individual fibres,
 - 5) fibres showing curvature.

INTERNATIONAL
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ISO
22262-1

First edition
2012-07-01



ISO 22262-1 – Morphology by PLM

7.2.3.7.1 Morphology

In general, for this part of ISO 22262, the presence of either the asbestiform or the non-asbestiform analogues of tremolite, actinolite, anthophyllite or richterite/winchite can usually be specified. If the majority of the amphibole fibres longer than 5 µm have aspect ratios equal to or lower than 5:1, and if the fibres do not exhibit any of the characteristics in c), it can be concluded that the amphibole is probably non-asbestiform, with the degree of certainty increasing with decreasing maximum aspect ratio. If any amphibole fibres longer than 5 µm with aspect ratios in the range of 20:1 or higher are observed, then it can be concluded that amphibole asbestos is probably present, with the degree of certainty increasing with increasing aspect ratio.

“amphibole asbestos probably present”: ANY “amphibole fibers longer than 5 µm with aspect ratios in the range of 20:1 or higher”

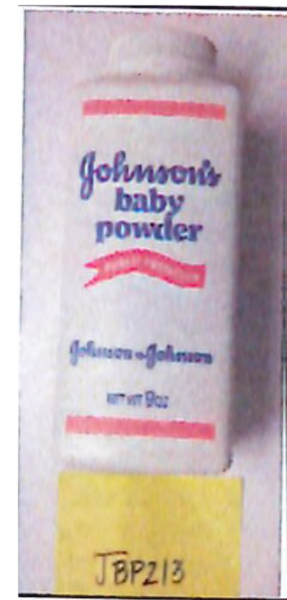
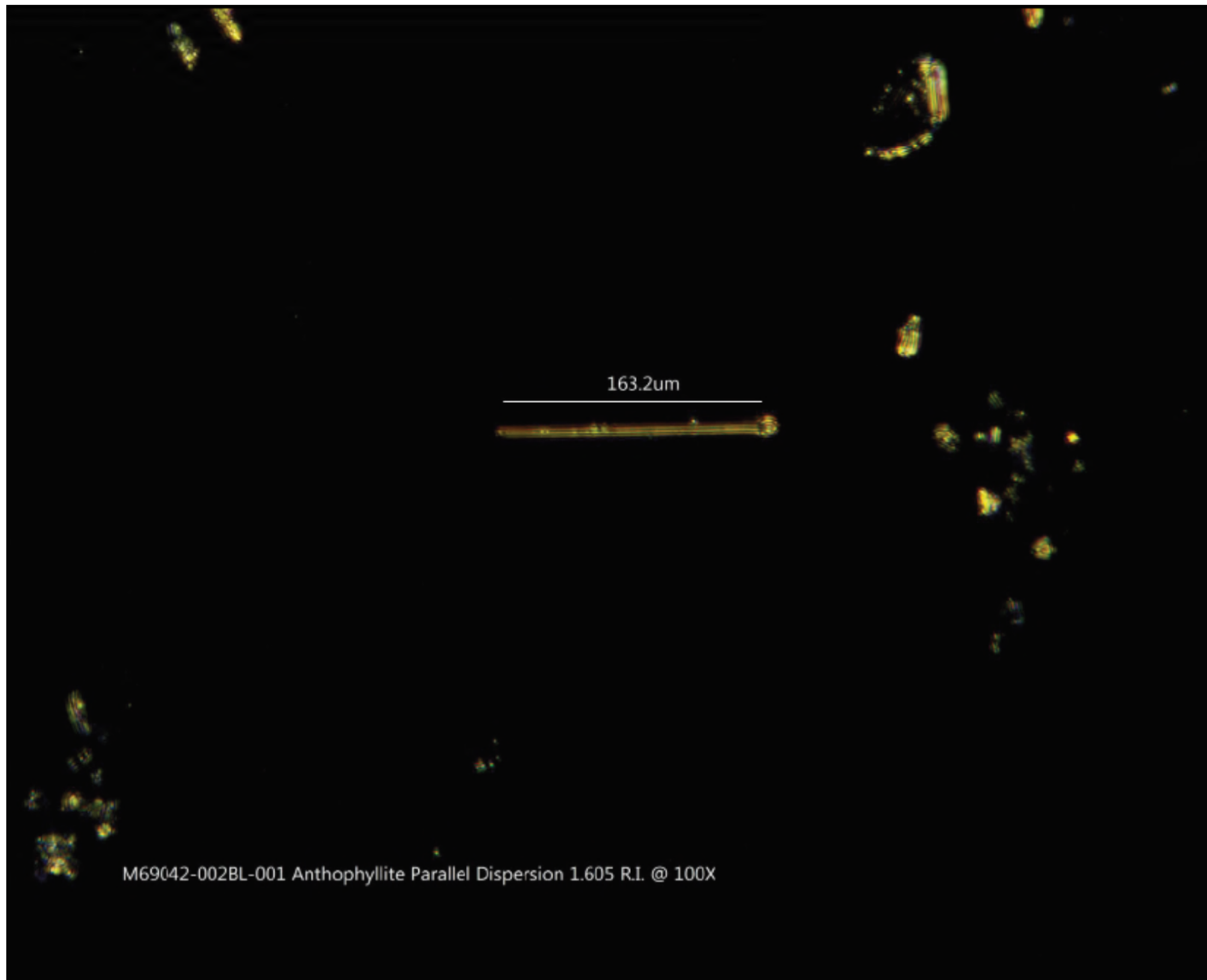


Tab 25, ISO 22262-1 Pg. 22-23

INTERNATIONAL
STANDARD

ISO
22262-1

First edition
2012-07-01



MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00867, Tab 9F



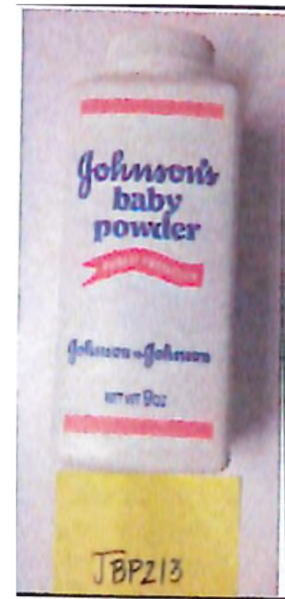
MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00868, Tab 9F

M69042-002BL-001 Anthophyllite Perpendicular dispersion



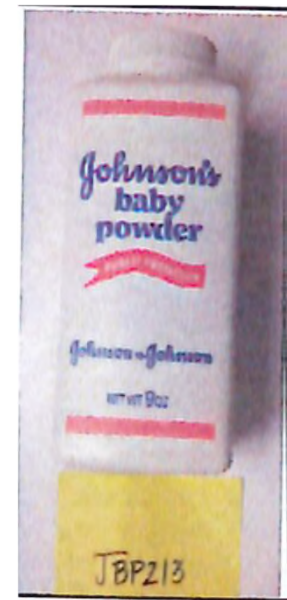
MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00869, Tab 9F

M69042-002BL-001 Anthophyllite Elongation @ 200X

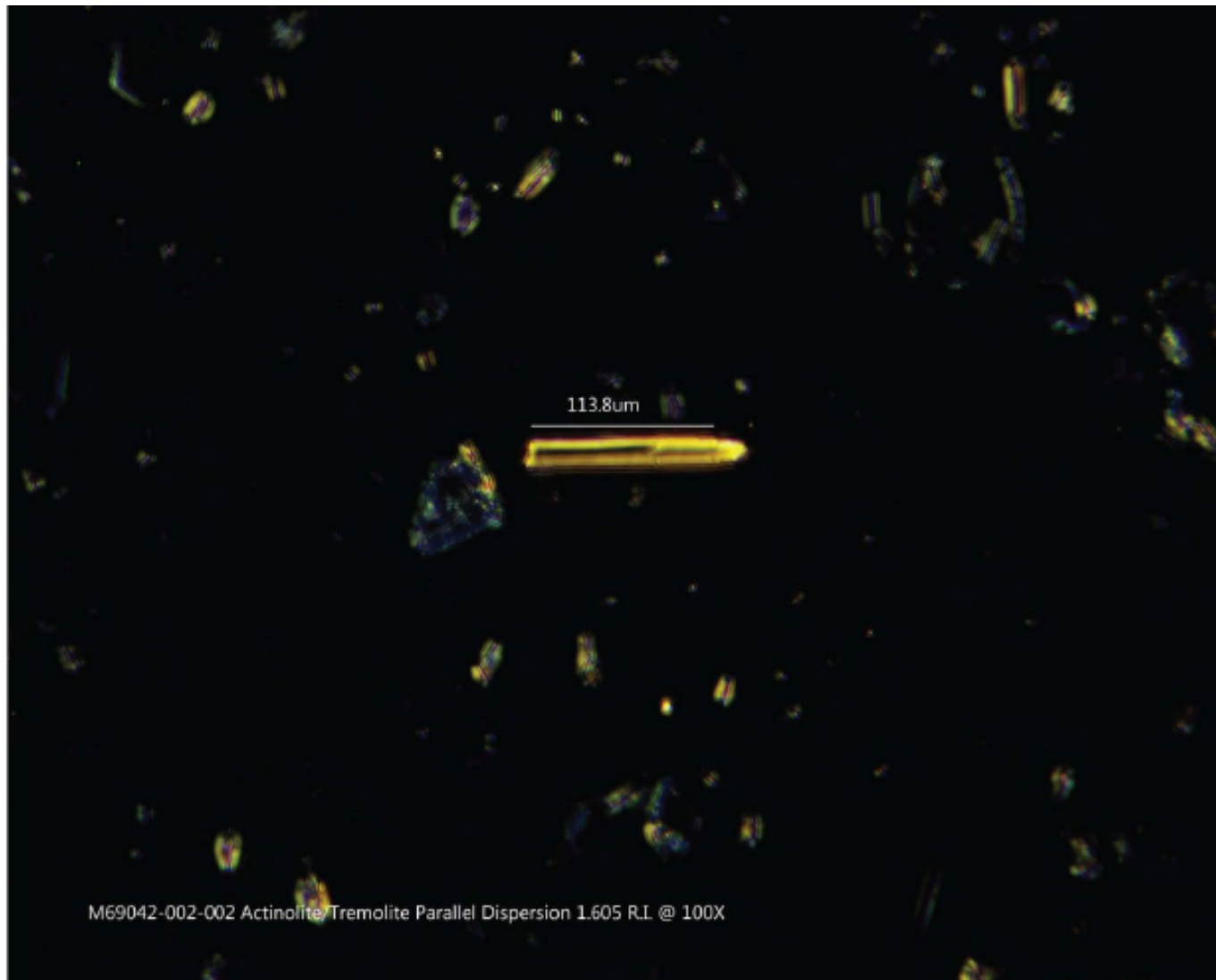


M69042-002BL-001 Anthophyllite Crossed Polars

MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00870, Tab 9F

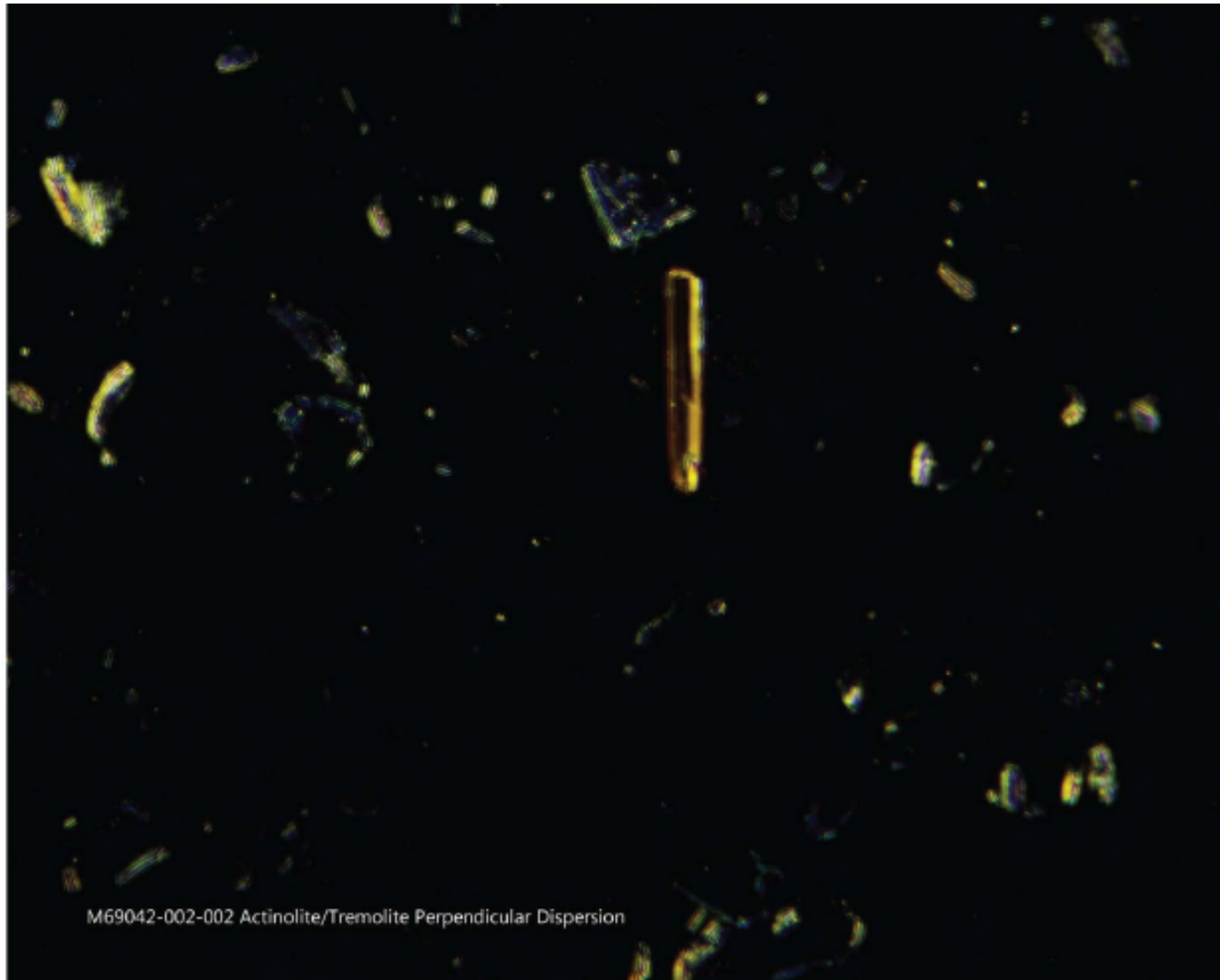


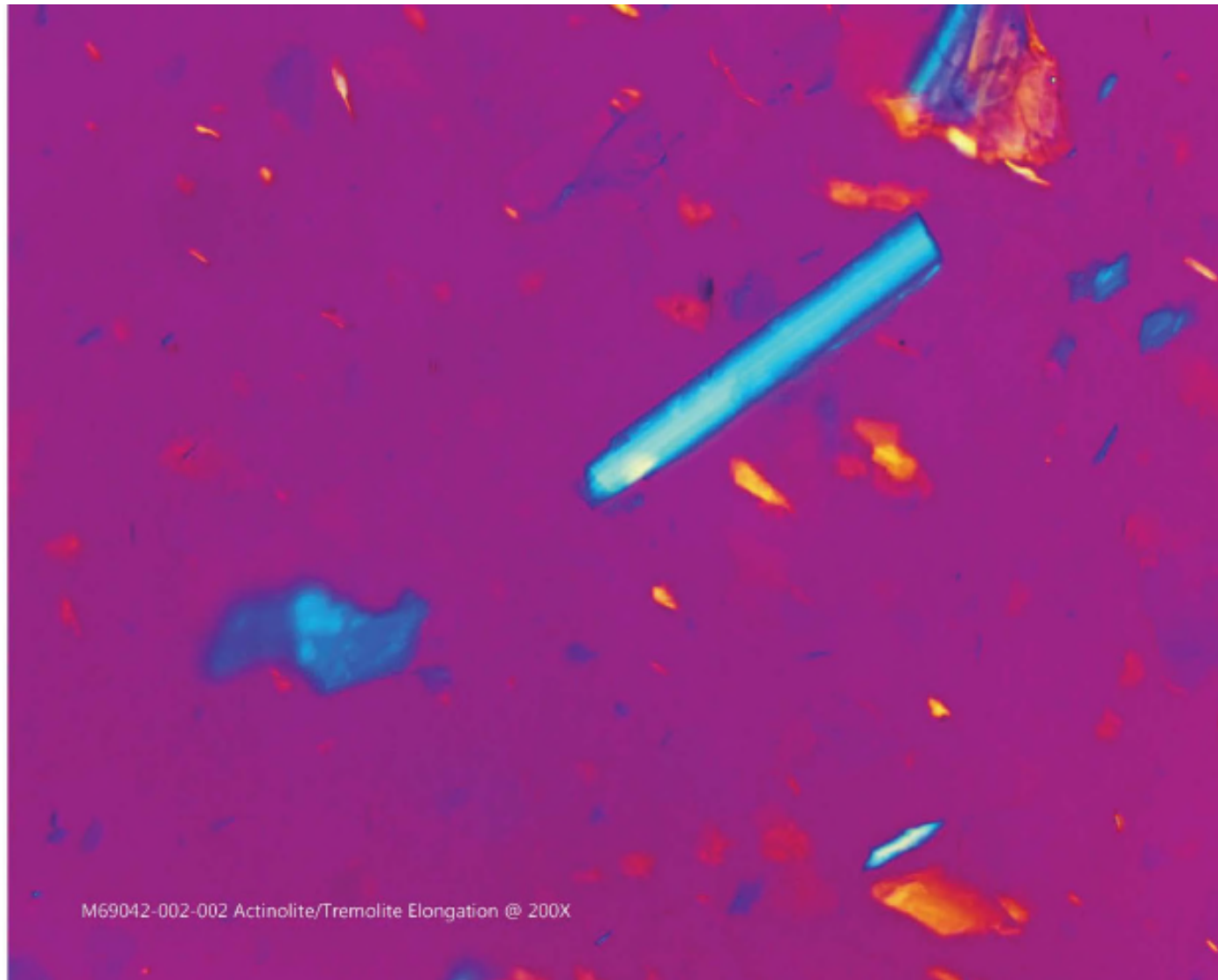
MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00859, Tab 9F



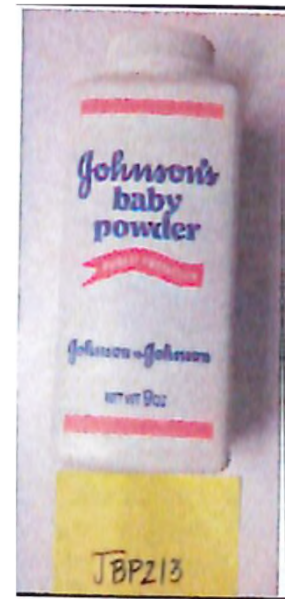


MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00860, Tab 9F





MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00861, Tab 9F



MAS 1/15/19 Report Backup Data
Binder,
Bates: Longo-MDL_00862, Tab 9F

M69042-002-002 Actinolite/Tremolite Crossed Polars



Example 2

Sample M69680-014BL

1983 S2S Sample



MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_01480-01506

Anthophyllite Asbestos, Actinolite/Tremolite Asbestos, and Fibrous Talc in 1983 S2S Sample

M69680-014BL-001 Act/Trem Parallel Dispersion 1.605 R.I. @ 100X

54.1um

<u>ASBESTOS MINERALS</u>	<u>EST. VOL. %</u>
Chrysotile.....	_____
Amosite.....	_____
Crocidolite.....	_____
Tremolite/Actinolite.....	< 0.1
Anthophyllite.....	< 0.1

Comments Actinolite/Tremolite and Anthophyllite asbestos observed. *** Moderate amount of fibrous Talc observed. X = Materials detected.

The method detection limit is 1% unless otherwise stated.

M69680-014BL-003 Anthophyllite Parallel Dispersion 1.605 R.I. @ 100X

73.7um

MAS: M69680-014BL

RJLG: 3149754

MAS 1/15/19 Report Backup Data Binder,
Bates: Longo-MDL_01481-1482, 01491,
Tab 9G

Results of MAS PLM Testing of J&J Products

MAS ISO-22262-1 Analysis

No Heavy Liquid Separation

The ISO 22262-1 PLM analysis showed that out of the 46 JBP/STS containers and 15 Imerys' railroad car samples analyzed by MAS, 20 containers (30 %) had detectable amounts of regulated amphibole asbestos, the rest were either non-detects or contained actinolite/tremolite cleavage fragments that had an aspect ratio of < 3:1.

Results for all 20 positive samples were found to contain <0.1 % asbestos. Also, for these positive ISO PLM samples, both regulated actinolite/tremolite and or anthophyllite asbestos were found.

PLM/Blount Method

Heavy Liquid Separation

The Blount/PLM method showed that out of the 71 historical JBP/STS containers and Imerys' railroad car samples analyzed by MAS, 41 (58%) had detectable amounts of regulated amphibole asbestos and the rest were either non-detects or contained only tremolite/actinolite cleavage fragments that had an aspect ratio that was less than 3:1.



Amphibole Asbestos detected in 41 of 71 (58%)

MAS Test Results: Consistent with Historical Testing Documents and Geology of Source Mines

Dr. Mark Krekeler, Ph.D.

- Professor, Miami University, Oxford, Ohio, Dept. of Geology

Tab 47, 2018 Krekeler Expert Report, Pg. 2, 31, 45

In summary, the reviewed documents have established the presence of asbestos in the talc mined and manufactured for Johnson's Baby Powder and Shower to Shower.

1. The presence of asbestos and fibrous talc was and is present in the mines Defendants' used for cosmetic talc;

Dr. Robert Cook, Ph.D.

- Former Professor, Auburn University, Dept. of Geology

Tab 48, 2019 Amended Cook Expert Report, Pg. 2, 42

Based on my review, it is my opinion to a reasonable degree of scientific certainty that the talc deposits that were used to source Defendants' talcum powder products (Italy, Vermont and China) contain fibrous talc, and chrysotile and/or fibrous amphiboles, all known human carcinogens. As to the Vermont talc deposits that sourced Defendants' talcum powder products, it

Amphibole Content of Cosmetic and Pharmaceutical Talcs

by A. M. Blount*

Environmental Health Perspectives
Vol. 94, pp. 225-230, 1991

**Sample I:
“tremolite
asbestos”**

Table 1. Counts of regulatory fibers in processed talcs.

Sample	Counts, particles/mg	SD	Particle shapes	Particles/FOV ^a
A	38	25	Cleavages	3/100
B	ND ^b			0/20
C	ND			0/20
D	< 25 ^c		Cleavages	0/20
E	ND			0/20
F	ND			0/20
G	ND			0/20
H	17	17	Cleavages and needles	2/20
I	226	59	Needles and fibers	17/20 ^d
	283	100	Needles and fibers	8/20
	291	98	Needles and fibers	9/20
	341	108	Needles and fibers	10/20
	102	51	Needles and fibers	3/20
J	25	14	Cleavages	1/20
	27	27	Cleavages	3/20
K	25	25	Cleavages	1/20
L	< 10 ^c		Needles	0/20
M	39	21	Cleavages and fibers	4/20
N	25	17	Prismatic pieces	3/20
O	ND			0/20

Tab 13, Blount 1991 article, Pg. 228

Sample I Was Johnson's Baby Powder Containing Windsor, Vermont Talc (Used from approx. 1967-2003)

A Italian

B Willow Creek,

C Pfizer, MT

D North Carolina

E Alabama

F Willow Creek, MT

G Barretts, MT (floated in Alabama)

H Italian

I Windsor, VT - baby powder

J Vermont

K Vermont

L Vermont

M Vermont

N Steetley, Ontario

O Montana Talc Co., MT

Key attached to
1992 Blount letter
to Luzenac

A - Italian

B - Montana - Willow Creek

C - Montana (Beamer Road) Pfizer

D - North Carolina

E - Alabama

F - Montana - Willow Creek

G - Montana Floated at Alabama
(Pfizer) Beamer?

H - Italian

I - Windsor - J & J JBP

J, K, L, M - other VT deposits - M = Troy

N - Timmins Ontario - Steetley

O - Willow Creek - Montana Talc Co

Key attached to J&J
File Copy of 1991
Blount Article

floated - one from
Whitcomb & Tracy

Tab 14, Bates: JNJNL61_000095946

Tab 15, Bates: JNJNL61_000014437

Alice M. Blount, Ph.D.
Mineralogist

GMW
April 23, 1998

M. Raymond Hatcher
MEHAFFY & WEBER

BEAUMONT, TEXAS

Dear Mr. Hatcher:

According to your letter of March 31, 1998, I have written and enclosed a report on the occurrence, regulation and up-to-date scientific view of asbestos, amphiboles and "intermediate" fibers. I have also enclosed copies of papers I have already have. The 1991 paper was written among industrial hygienists that indicate talc because there was a regulation in the case and wanted to set the record

Although my papers report an improved method for analysis, the determinations for the sample labeled I (Johnson & Johnson's Vermont talc) have been done by the traditional methods as well (see Table 2, page 567 in the 1990 paper). As I told you, I believe that Johnson & Johnson's Vermont talc contains trace amounts of asbestos which are well below those specified by OSHA. It should be noted that the proposed FDA regulation, which was never finalized, also

Although my papers report an improved method for analysis, the determinations for the sample labeled I (Johnson & Johnson's Vermont talc) have been done by the traditional methods as well (see Table 2, page 567 in the 1990 paper). As I told you, I believe that Johnson & Johnson's Vermont talc contains trace amounts of asbestos which are well below those specified by OSHA. It should be noted that the proposed FDA regulation, which was never finalized, also

I may be away for short periods during the coming weeks, but I do check for messages on my work phone at the number you have been using.

Sincerely yours,

Alice M. Blount

Alice M. Blount, Ph.D.

Box 1437
Rutland, VT 05701
Phone: 802-747-4857
e-mail: ambblount@together.net

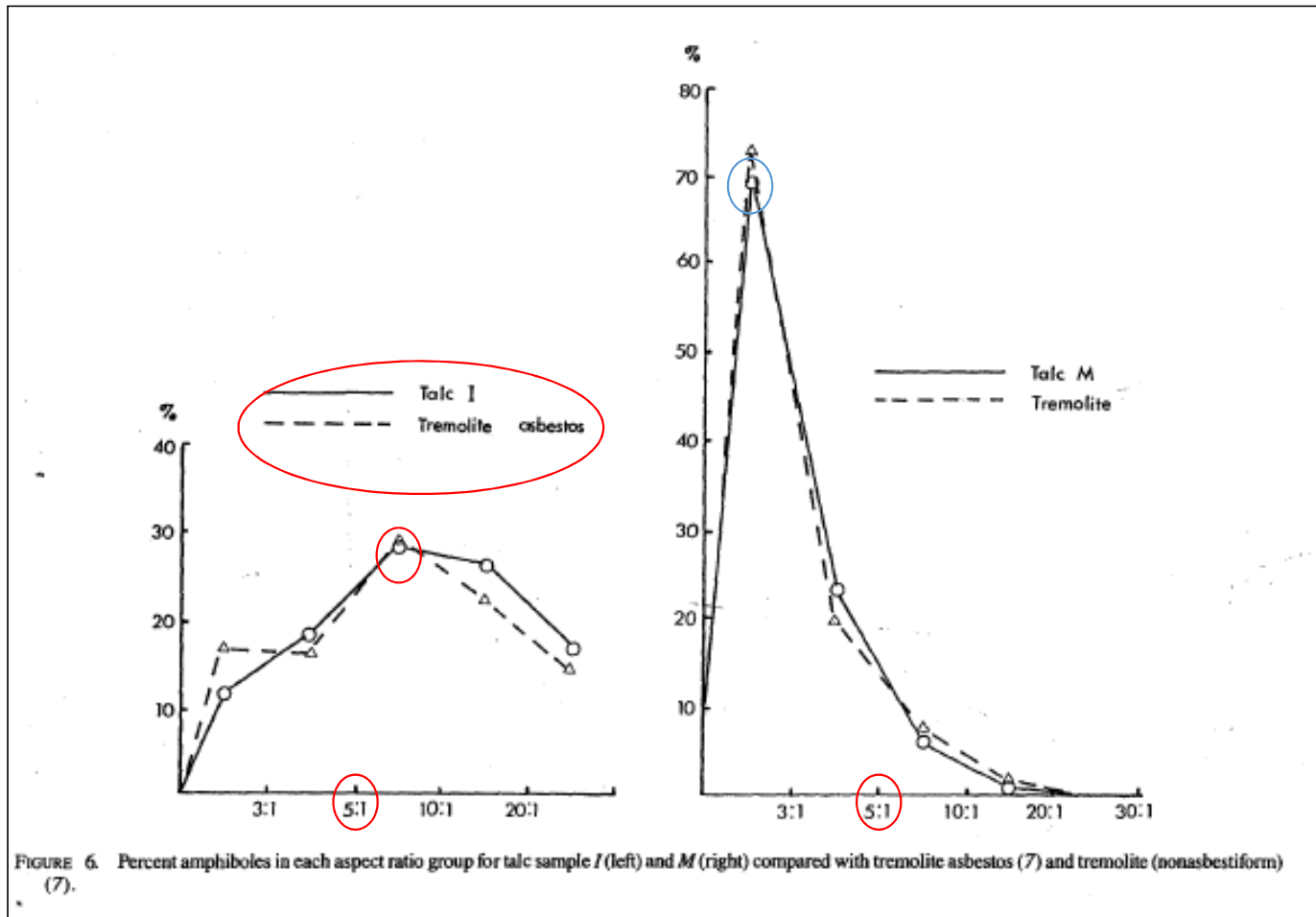
Alice M. Blount

Alice M. Blount, Ph.D.

Tab 16, 4/23/98
letter from Blount
to Hatcher

J&J-0049150
JNJNL61_000052427

Blount 1991: Tremolite Asbestos in JBP Consistent with Campbell



7. Campbell, W. J., Blake, R. L., Brown, L. L., Cather, E. E., and Sjoberg, J. J. Selected Silicate Minerals and Their Asbestiform Varieties. U.S. Bureau of Mines Information Circular 8751: 0-56, Pittsburgh, PA, 1977.

IARC: Reliance on 1991 Blount Study Finding Asbestos in “one sample” (Sample I - JBP)


Blount (1991) examined pharmaceutical- and cosmetic-grade talcs for asbestiform amphibole content using a density-optical method. High-grade talc product samples ($n = 15$) were collected from deposits in Montana, Vermont, North Carolina, Alabama, and from outside the USA but available in the US market. Samples were uniformly low in amphibole content (with counts in the range of 0–341 particles/mg), and some samples appeared to be completely free of amphibole minerals. In samples containing amphibole minerals, cleavage-type and asbestos-type minerals were observed. Only one sample was found to contain an amphibole particle size distribution typical of asbestos.

Forensic Analytical Finding of Anthophyllite Asbestos in JBP by TEM

Tab 30, J&J 1/24/04 Fax with attached report,
Bates: JNJI4T5_000004099

To: Steve Mann - CPC	Fax: (908) 904-2738
From: Marc Monseau	Date: 2/24/2004
Corporate Communications	
Re: Asbestos	Pages: 4

ANALYTICAL RESULTS						
Client Sample Number	Lab Sample Number	Organic Weight Percent	Acid-Soluble Weight Percent	Asbestos Weight Percent	Asbestos Type(s)	Residue Weight Percent
TEM-30 (Johnson's baby powder)	20025738	3.8%	8.7%	0.20%	AM	80.3%
TEM-33 (Revlon blush)	20025739	78.7%	13.1%	<0.0001%	ND	57.2%

 Forensic Analytical QUANTITATIVE ANALYSIS REPORT ASBESTOS IN BULK MATERIAL Transmission Electron Microscopy	
Michael Bowler 4088 Alva Ct Piscataway, NJ 08854	Page: 1 of 1 Client Number: A30388-1 Report Number: T008825 Date Received: 12/19/03 Analyst: RE Date Analyzed: 1/5/04 Data Reported: 1/5/04
Date Collected: Job ID: KCRA Television/Dave Walker Site:	


Date received 2003 =
Vermont Talc

Mark R. Floyd, EM Supervisor, Hayward Laboratory

* EPA Test Method 600/4-83/116, Part 2.5; Method for the Determination of Asbestos in Bulk Building Materials.

** Asbestos types: CH-chrysotile; AM-amosite; TR-tremolite; AC-actinolite; CR-crocidolite; AN-anthophyllite; ND-none detected.

Hammondsville Cosmetic Ore ("HC") used by J&J

Attachment 5		Page 1 of 2
		WINDSOR MINERALS INC.
<u>CONFIDENTIAL</u>		Windsor, Vermont
Subject: Concerning a 6 month ore study on <u>asbestiform content.</u>		January 14, 1975

The samples represented both the industrial materials produced at the Gassetts (GI) and West Windsor (WI) mill sites and the ores used in the cosmetic production (HC).

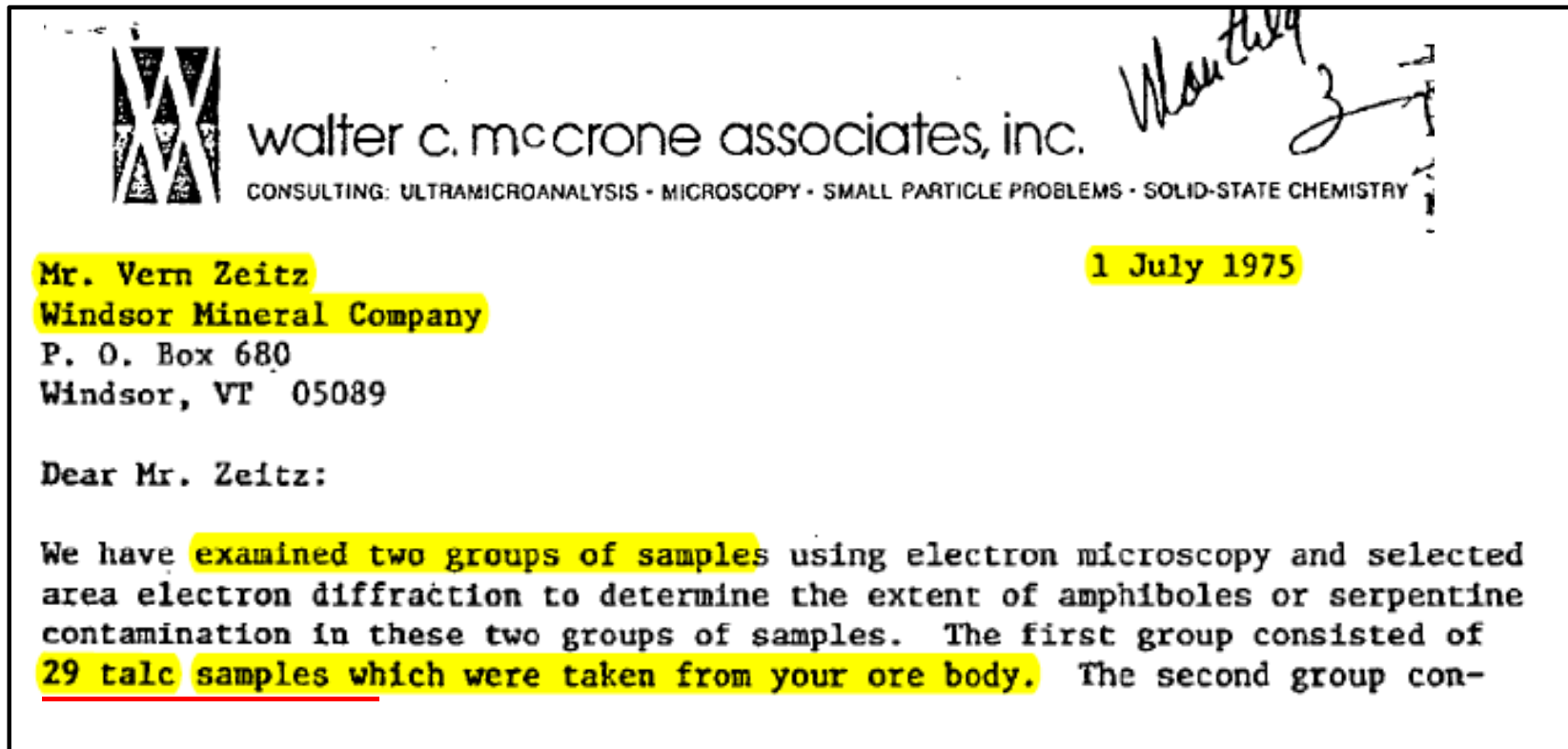
GI = Gassetts

WI = West Windsor

HC = Hammondsville Cosmetic

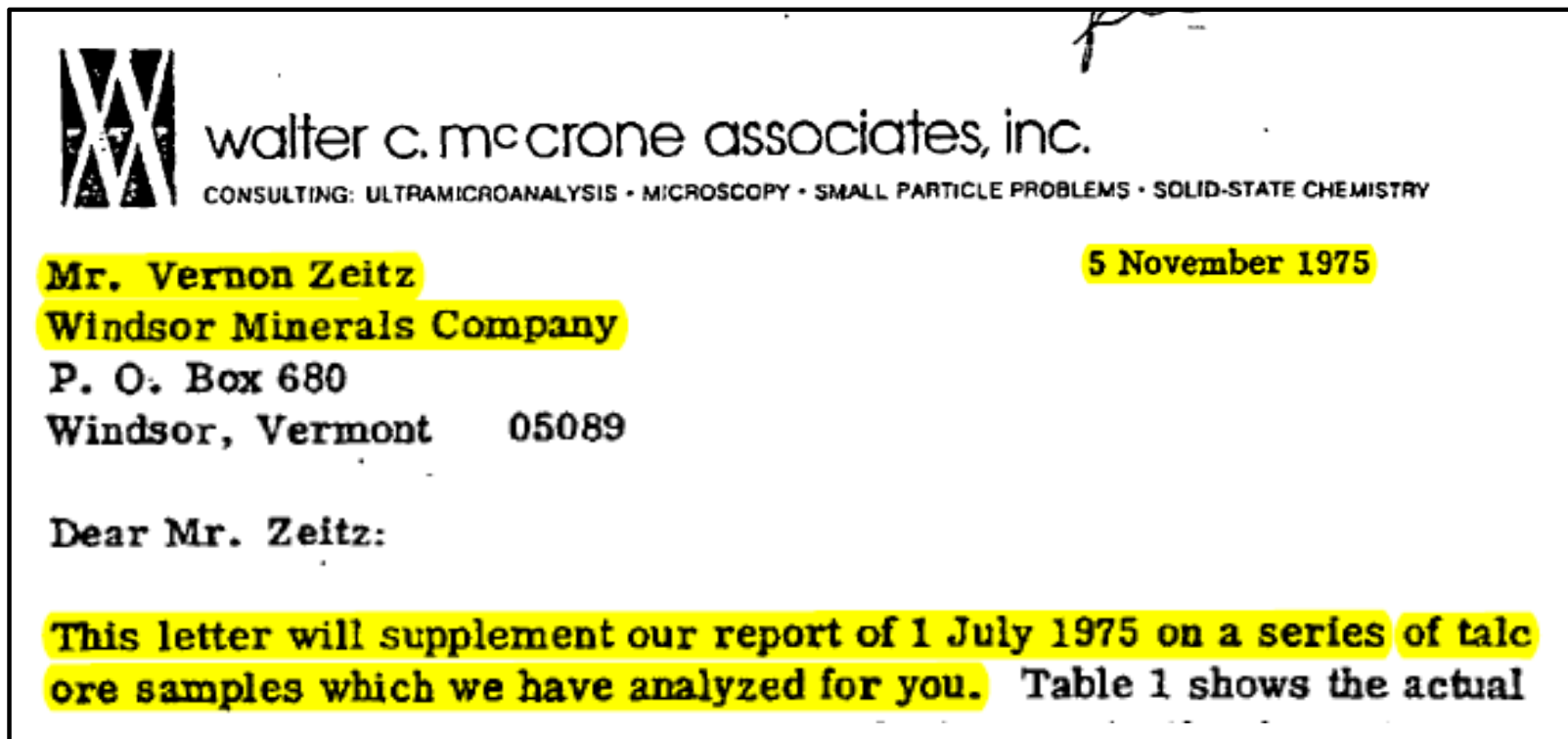
Tab 34, 1/14/75 Windsor
Minerals Memo,
Bates: JNJMX68_000002659

Asbestos in "HC" Hammondsville Cosmetic Talc



Tab 35, 7/1/75 letter from Grieger to
Zeitz, Bates: JNJMX68_000012745

Asbestos in “HC” Hammondsville Cosmetic Talc



Tab 36, 11/5/75 letter from Grieger to Zeitz,
Bates: JNJNL61_000079334

July 1975 Report

Sample No.	Confirmed asbestos, visual
W-GI	0
BI-GI	0
BI-WI	0
FI-WI	Low
Y-GI	0
W-HC	0
V-HC	0
Z-GT	0
Y-HC	0
DI-HC	0
GI-HC	0
X-HC	Low
FI-HC	Medium
V-WI	0
V-GI	Low
EI-HC	Low
GI-WI	Low
CI-HC	0
DI-GI	0
CI-GI	Low
U-GI	0
H1-HC	0
H1-WI	0
BI-HC	Low
E1-GI	0
A1-HC	0
E1-WI	0
Z-HC	Low
DI-WI	0

GI = Gassetts

WI = West Windsor

HC =
Hammondsville
Cosmetic

November 1975 Report

Sample Number	Date	Fibers of Asbestos	
D-HC	7/22/74	7/26/74	
D-GI	7/15	7/29	
F-HC	9/13	9/7	
H-WI	9/16	9/23	
I-WI	9/23	9/28	
P-GI	10/28	11/1	
Q-HC	11/4	11/8	
U-HC	12/2	12/6	
U-GI	12/2	12/6	0
V-WI	12/9	12/20	0
V-HC	12/9	12/13	0
V-GI	12/9	12/16	2 amph.
W-HC	12/16	12/20	0
W-GI	12/16	12/20	0
X-HC	12/26	12/28	2 amph.
Y-HC	12/30	1/3/75	0
Y-GI	12/30	1/6/75	0
Z-HC	1/6/75	1/10/75	9 amph.
Z-GI	1/6	1/13	0
A1-HC	1/13	1/17	0
B1-HC	2/24	2/28	5 amph.
B1-WI	2/24	3/7	0
B1-GI	2/24	3/3	0
C1-HC	3/3	3/7	0
C1-GI	3/3	3/10	6 amph.
D1-HC	3/10	3/14	0
D1-WI	3/10	3/14	0
D1-GI	3/10	3/17	0
E1-HC	3/17	3/21	2 amph.
E1-WI	3/14	3/21	0
E1-GI	3/17	3/24	0
F1-HC	3/24	3/29	10 amph.
F1-WI	3/24	3/29	1 amph.
			1 antigorite
G1-HC	3/31	4/4	0
G1-WI	3/31	4/4	1 amph.
H1-HC	4/7	4/11	0
H1-WI	4/7	4/11	0
D -WI	7/15	8/2	
H -GI	9/16	9/23	

Fibers of Asbestos

0

0

0

2 amph.

0

0

2 amph.

0

0

9 amph.

0

0

5 amph.

0

0

0

6 amph.

0

0

0

2 amph.

0

0

10 amph.

1 amph.

1 antigorite

0

1 amph.

0

0

Tab 36

Tab 36

Tab 35

July 1975 Report –Asbestos “Fibers” and “Bundles”

TABLE 3	
Sample content, based on photomicrographs	
DI-HC	Blocky talc, 2 silicate fibers
X-HC	2 amphiboles, 1 talc hard
FI-HC	2 bundles of amphiboles, 2 single amphibole fibers
V-WI	2 silicates
V-GI	2 amphibole and 1 amphibole-like fiber without diffraction pattern
GI-WI	2 talc ribbons, fine particulate contamination and organic crud
CI-HC	blocky talc, talc fibers, silicates and 1 amphibole
W-GI	bacteria, silicates, blocky talc and organic fibrils
BI-GI	rolled talc, organic fiber and talc ribbons
BI-WI	silicates and talc ribbons
Y-GI	blocky talc, crystalline square particles
FI-WI	some organic material, fine crystalline particles about 500 Å in size and silicates
W-HC	large particles, 1 amphibole, 1 fibrous antigorite, silicates and rolled talc
Y-HC	blocky talc and organic material
V-HC	silicates
BI-HC	organic material
H-WI	rolled talc fibers, blocky talc and 2 amphibole bundles
U-GI	lots of organic material, 1 amphibole
CI-GI	organic material, blocky talc and silicates
DI-GI	silicates, talc ribbon, fibrous talc, blocky talc, organic fibers and 2 bundles of amphibole
CI-HC	blocky talc, organic material, rolled talc and silicates
V-WI	1 amphibole and fibrous talc
Z-HC	silicates
DI-WI	small square particulate matter about 1000 Å, 3 bundles of amphibole
AI-HC	1 amphibole, fine particles, fibrous talc and blocky talc
EI-WI	silicates
D-HCS*	blocky talc
U-GIS	1 bundle of silicates and blocky talc
Z-HCS	blocky talc
X-HCS	1 bundle which looks like amphibole, no diffraction pattern available
DI-HCS	silicates
W-GIS	fine particles
V-HCS	1 rolled talc, 1 amphibole and 1 silicate
D-HIS	clean
D-GIS	silicates and talc ribbons
FNCS	organic fibrils
I-WIS	clean
P-GIS	blocky talc and 2 silicates
Q-HCS	1 bundle of amphiboles, 1 blocky talc fiber
FI-WIS	clean
DI-GIS	clean
Y-HCS	some blocky fibrous talc

Luzenac 2002 – Tremolite Asbestos from Argonaut Vermont Talc Mine



Luzenac America Technical Center • 8985 East Nichols Avenue • Englewood, CO 80112 • (303) 643-0451 • Fax: (303) 799-8926

TECHNICAL REPORT

To: David Crouse
Analytical Project No: **A01709**
Date: **23-May-02**

From: Julie Pier
Analytical and Technical Support

Copy: J. M. Godla
S. S. Mauney
R. J. Zazenski

Subject: **ANALYSIS OF FIBROUS MATERIAL FROM ARGONAUT
WASTE ROCK**

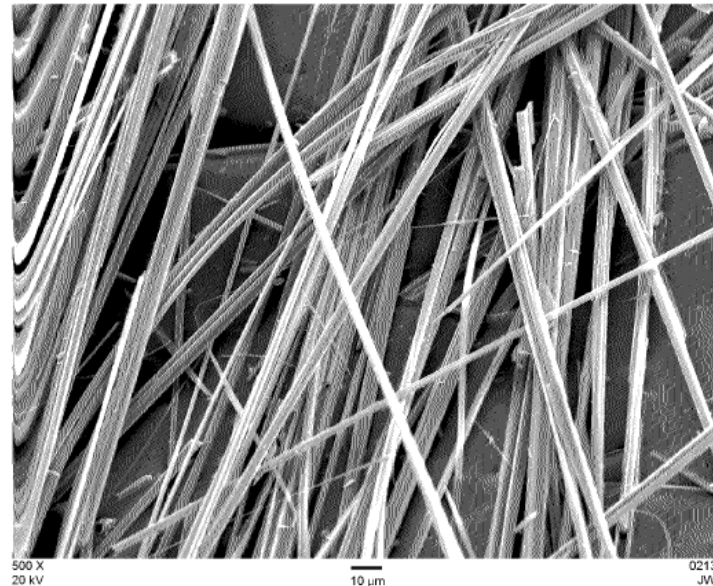
Results:

The fibrous material is tremolite.

ANALYSIS OF FIBROUS MATERIAL FROM ARGONAUT WASTE ROCK Project No. A01709

Plate 1

LUZENAC AMERICA TECHNICAL CENTER
23-May-02
J.W. Pier



SEM IMAGE
Fibrous material found in
Argonaut waste rock identified as
tremolite. The material clearly
has an extremely high aspect
ratio.

Tab 37, Luzenac 5/23/02 Testing Memo,
Bates: IMERYs 422289-422290

Luzenac 2002 – Tremolite Asbestos from J&J's Argonaut Vermont Talc Source Mine



Luzenac America Technical Center • 8985 East Nichols Avenue • Englewood, CO 80112 • (303) 643-0451 • Fax: (303) 799-8926

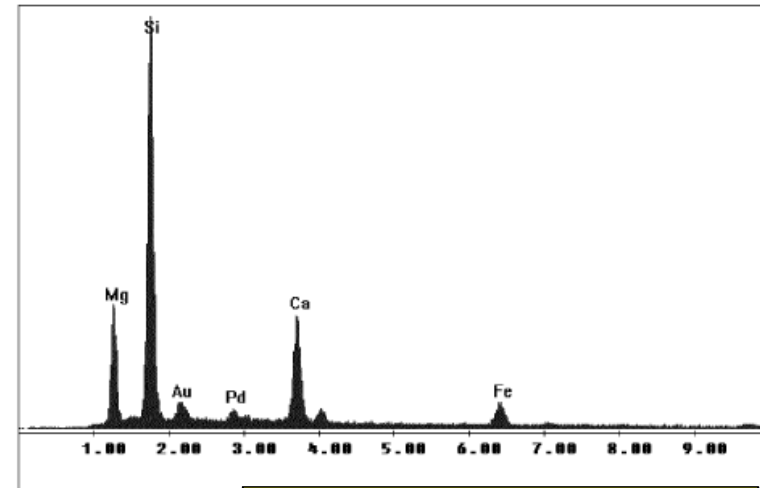
TECHNICAL REPORT

To: David Crouse
Analytical Project No: A01709
Date: 23-May-02

From: Julie Pier
Analytical and Technical Support

Copy: J. M. Godla
S. S. Mauney
R. J. Zazenski

Subject: ANALYSIS OF FIBROUS MATERIAL FROM ARGONAUT
WASTE ROCK



EDS CHEMICAL ANALYSIS
The chemical analysis of the material, above, is consistent with tremolite.

Au and Pd peaks are from a conductive coating applied for SEM analysis.

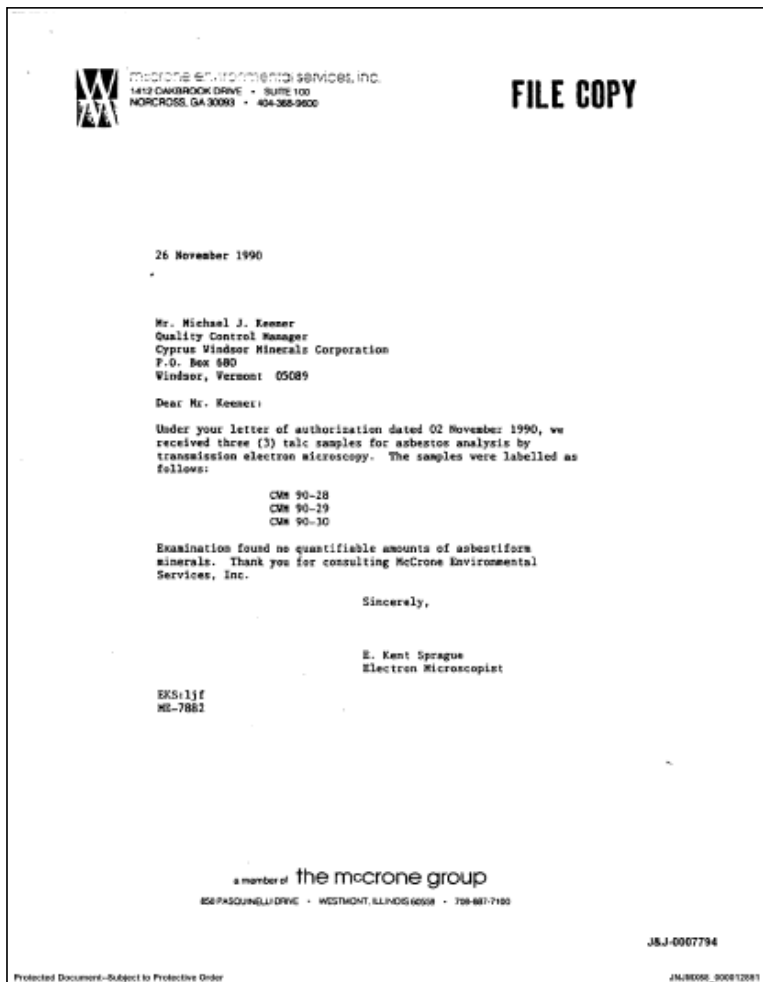
EDS (EDXA) Spectra

Results:

The fibrous material is tremolite.

Tab 37, Luzenac 5/23/02 Testing Memo,
Bates: IMERYS 422289-422290

McCrone 1990 - Asbestos in Vermont Talc – Cover Letter Says “No Quantifiable Amounts”



Under your letter of authorization dated 02 November 1990, we received three (3) talc samples for asbestos analysis by transmission electron microscopy. The samples were labelled as follows:

CMH 90-28

CMH 90-29

CMH 90-30

Examination found no quantifiable amounts of asbestos-form minerals. Thank you for consulting McCrone Environmental Services, Inc.

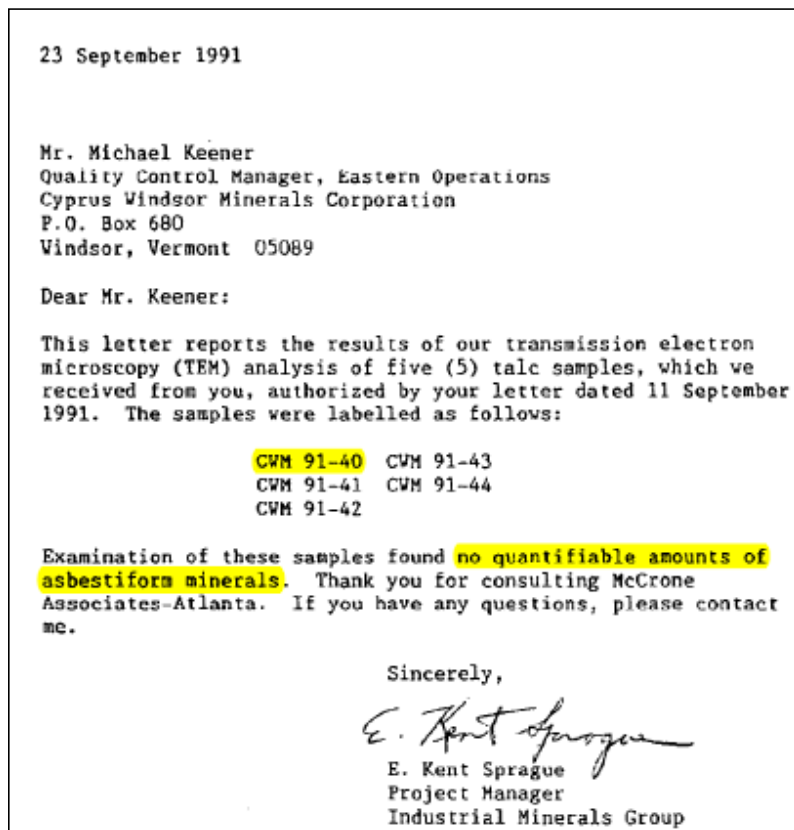
Tab 38, 1990 McCrone Testing, Bates: JN/JMX68_000012852

Tab 38, 1990 McCrone
Testing,
Bates: JNJMX68 000012854

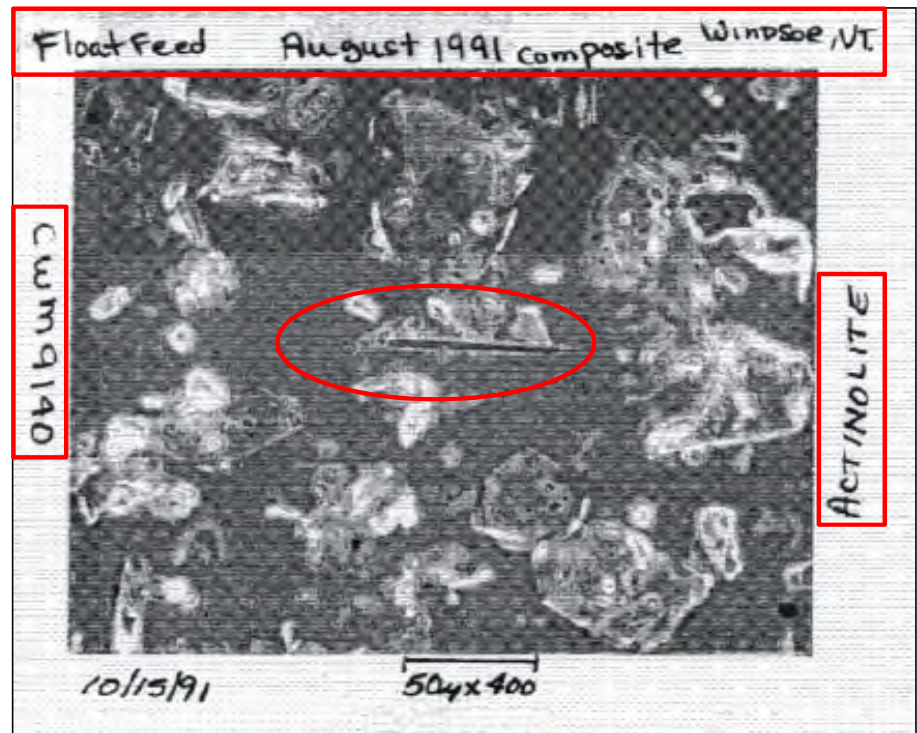
[illegible]

Cyprus 1991 – Photographs Showing Asbestos when McCrone Cover Letter Says “No Quantifiable Amounts”

McCrone Reports



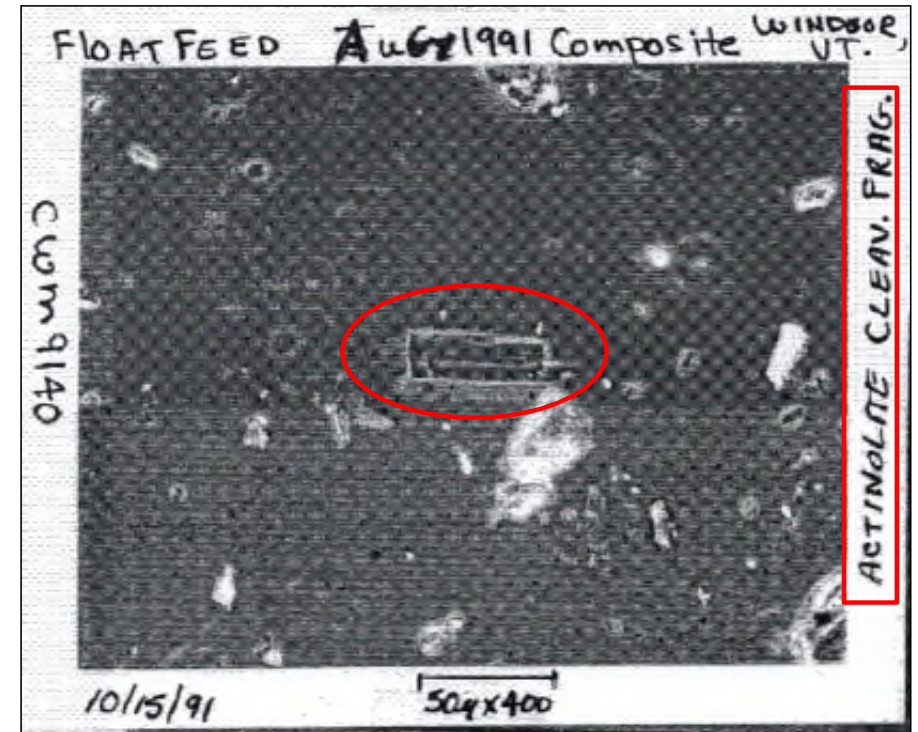
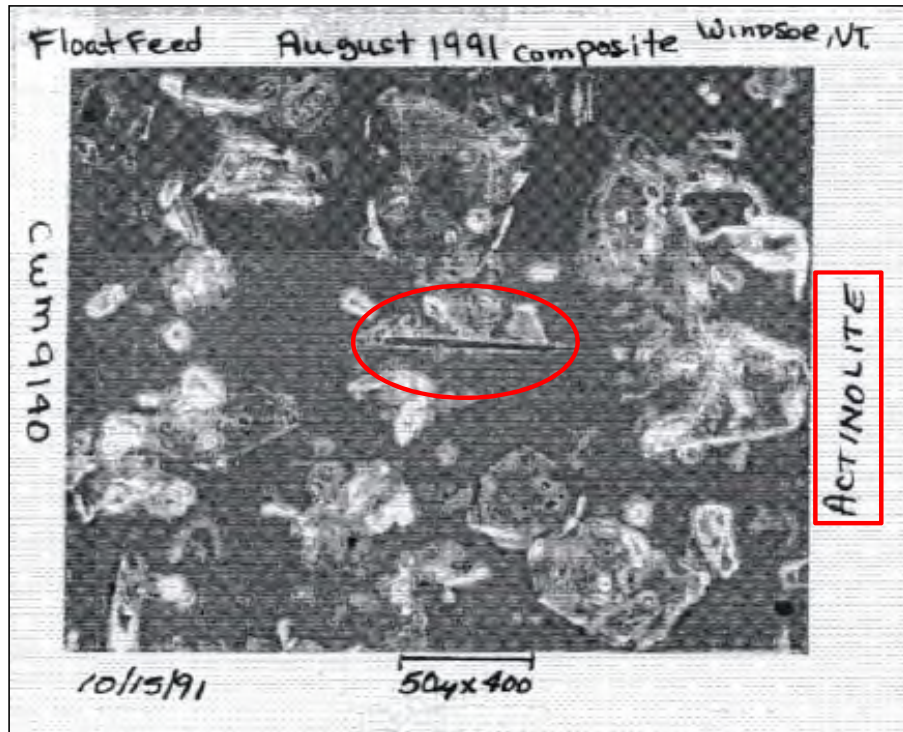
Cyprus Testing



Tab 39, 1991 McCrone Testing, Bates: JNJ 000280852

Tab 40, 1991 Cyprus Minerals Testing, Bates: AB-CYP-41-0001586

Cyprus 1991 – Distinguishing Between Actinolite Asbestos and Actinolite “Cleavage Fragments”



J&J's Italian Talc Source - Fibrous Tremolite (asbestos)

Grit consists of that portion of ground talc which is angular, or oversize, particularly in thickness. Grit includes both oversize and nonplaty talc particles as well as mineral contaminants. It occurs as aggregates of talc and contaminants, as acicular and fibrous particles of talc and amphibole, as shards and granules of amphibole or carbonate, and as prismatic grains of titanite, rutile, zircon, apatite, and other accessory minerals.

The Italian No. 1 talc contains from less than 1 per cent to about 3 per cent of contaminants. The contamination is natural and consists mostly of carbonate with minor amphibole and rare accessory minerals. The carbonate component has been identified petrographically as primarily dolomite ($\text{CaO} \cdot \text{MgO} \cdot 2\text{CO}_2$) plus a minor amount of probable magnesite ($\text{MgO} \cdot \text{CO}_2$). No calcite ($\text{CaO} \cdot \text{CO}_2$) was identified. The amphibole component has been established to be the variety tremolite ($2\text{CaO} \cdot 5\text{MgO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$).

BATTELLE MEMORIAL INSTITUTE

Tab 41A, 1958 Battelle Report,
Bates: JNJAZ55_000000912

J&J Vermont Talc Source – Fibrous Tremolite and Actinolite (asbestos)

CYPRUS ORE RESERVES - ARSENIC & TREMOLITE

Excerpts from Cyprus Talc Reserve Report by R.C. Munro

DATE March 25, 1992

Geology & Environment

There are some important environmental issues related to the geology and mineralogy of the Cyprus talc deposits, particularly in Vermont.

Tremolite

The other serious mineralogical contaminant in the talc ores of Vermont is the fibrous variety of the amphibole minerals, tremolite and actinolite (hydrous calcium iron-magnesium silicates) which have been classified as asbestiform minerals by OSHA and EPA. OSHA was expected to de-classify non-fibrous (blocky) tremolite on February 29, but has not as yet announced their decision.

J&J Vermont Talc Source – Fibrous Tremolite and Actinolite (asbestos)

CYPRUS ORE RESERVES - ARSENIC & TREMOLITE

Excerpts from Cyprus Talc Reserve Report by R.C. Munro

DATE March 25, 1992

Vermont talcs are derived from altered serpentine - a natural host for asbestiform minerals. There is certainly visible tremolite and actinolite in specific zones of the Vermont deposits - fibrous tremolite was identified by the writer in exposures and cores at the East Argonaut and Black Bear mines. Cyprus staff report past tremolite from the Hammondsvile and Clifton deposits.

Dr. Wylie's Proposed Definition of Asbestos (EPA 600/R-93)

The U.S. Environmental Protection Agency (EPA) polarized light microscopy (PLM) method (1993) specifies the characteristics of asbestos, summarized as follows: means aspect ratio (length/width) ranging from 20:1 to 100:1 or higher for fibers longer than 5 micrometers, very thin fibrils, usually less than 0.5 micrometers in width, and 2 or more of the following properties: fibers occurring in bundles, bundles displaying splayed ends, matted masses of individual fibrils and fibers showing curvature. These characteristics are evident in the asbestos shown in Figure 6(a).

USEPA (1993) Test Method: Method for the determination of asbestos in bulk building materials. Perkins RL and Harvey BW June 1993, *EPA/600/R-93/116*
<https://www.nist.gov/sites/default/files/documents/nvlap/EPA-600-R-93-116.pdf>.

EPA 2006: R-93 Does Not Apply to Asbestos That is a Natural Contaminant (i.e., noncommercial amphiboles)



The R. J. Lee Report further states that EPA's data inflated the asbestos fiber count by ignoring the Agency's own "definition" of asbestos. To support this claim, the R.J. Lee Report cites the glossary of "Method for Determination of Asbestos in Bulk Building Materials", EPA 600/R-93/116, 1993, which states, in part, "With the light microscope, the asbestiform habit is generally recognized by the following characteristics: Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 microns." The building material analytical method is designed to detect commercially processed asbestos in items like floor tiles, roofing felts, paper insulation, paints, and mastics, not naturally occurring asbestos on air filters or in soil samples. To present the 20:1 aspect ratio for commercial grade asbestos as a universal EPA policy, and to advocate its use as an appropriate standard for analyzing air samples of naturally occurring asbestos is inappropriate and contradictory to use of the PCME dimensional criteria as a tool for assessing exposure risk.

EPA 2006: Asbestos Structures are Identified According to Test Protocols “Regardless of the Manner By Which They Were Formed”

Cleavage fragment is a geologic term which refers to structures that form when non-fibrous forms of asbestos minerals split along crystallographic planes, as opposed to asbestos fibers which form from crystalline growth. The R.J. Lee Report maintains that there is a toxicological difference between asbestos structures which formed as fiber crystals and fibers which formed by cleavage plane separation. Page 3 of the R.J. Lee Report states that cleavage fragments are “not known to produce asbestos-like disease.”

It is the position of EPA, the U.S. Centers for Disease Control and Prevention, Agency for Toxic Substances and Disease Registry (ATSDR) and National Institute for Occupational Safety and Health (NIOSH), and the American Thoracic Society, among others, that microscopic structures of amphibole and serpentine minerals that are asbestiform and meet the size definition of PCM fibers, should be counted as asbestos, regardless of the manner by which they were formed. There are four

Tab 43, EPA 4/20/2006 report, pg. 11

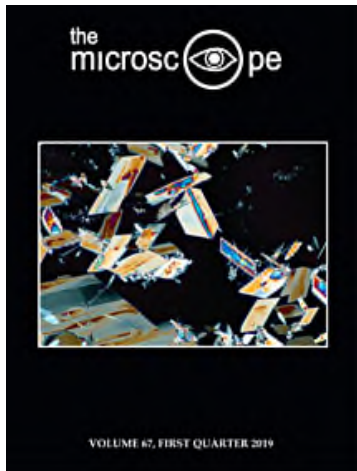


The Wylie/R-93 Criteria Would Result in Asbestos Being Misclassified as “Non-Asbestos”

THE MICROSCOPE • Vol. 63:1, pp 11-20 (2015)

Procedure for the Analysis of Talc for Asbestos

James R. Millette, Ph.D., D-IBFES
Millette Technical Consulting¹



ther way. Research by Wylie (25) reported in 1985 showed that 50% of the fibers in a known amosite (grunerite) asbestos sample would not be counted if a 20:1 aspect ratio were used as a criterion. Compari-

Tab 22, Millette 2015, Pg. 16

The Wylie/R-93 Criteria Would Result in Asbestos Being Misclassified as “Non-Asbestos”

This tremolite asbestos structure aspect ratio is consistent with the NIST tremolite asbestos standard, Blount’s tremolite asbestos findings for the off the shelf cosmetic talc container she tested, Campbell’s milled tremolite asbestos and Langer & Nolan’s published tremolite asbestos aspect ratio of 10.9 to 1. In the Blount publication, it was reported that the average aspect for non-asbestiform tremolite (cleavage fragments) was approximately 2:1.

Asbestiform tremolite/anthophyllite aspect ratio summary is as follows:

1. MDL ATEM analysis:	: 11:1	→	MAS Testing of J&J Talc
2. Blount	: 9:1		
3. Campbell	: 9:1		
4. Langer	: 11:1		
5. J&J 3/11/2018	: 10:1	→	MAS Testing of J&J Talc
6. NIST 1875 Tre. Std.	: 10:1		

Exhibit 9

FILE copy

CONFIDENTIAL

MARCH, 1974

To: Windsor Minerals Inc., Windsor, Vermont 05089

From: R.C. Reynolds Jr., Department of Earth Sciences,
Dartmouth College, Hanover, New Hampshire 03755

Subject: Analysis of Talc Products and Ores for Asbestiform
Amphiboles

INTRODUCTION:

The purpose of this study is to develop methods for measuring the concentration of asbestiform amphiboles in fine-grained talc products and talc ores. In principle, the problem might be simply solved by the microscopic counting of amphibole grains in samples that are suspended in oils of suitable refractive index, so that amphiboles are optically emphasized with respect to talc. In practice, however, grain counting is valid only if the dimensions of each grain are measured. This requirement arises from the large particle size range present, and from the wide variation of aspect ratios among the amphibole grains (see plate 16). In any event, grain-counting methods are inapplicable to whole samples because an inordinate number of grains must be considered. This makes the analysis time prohibitively long. For example, if a concentration of 100 ppm amphibole is assumed, and if 100 amphibole grains is the acceptable minimum that provides good statistical data, then one million grains must be considered. This requirement would be

easily met if the amphiboles were easily distinguished from other minerals. But even in suitable refractive index oils, many grains are ambiguous and require manipulations in order to verify their identity. Such ambiguous grains are (1) fibrous talc and carbonate grains in certain orientations (plate 21), (2) inclusion-filled grains (plate 21), (3) broken amphibole grains that have equant shapes (plate 9), and (4) grain aggregates (plate 20). If only 1% of the grains are ambiguous, and experience indicates that this is a conservative figure, then the example cited above would demand that attention be paid to 10,000 grains in a single sample.

For the reasons described above, a concentration technique is mandatory because it brings the amphiboles into a reasonable concentration range for optical or other methods of analysis. Such a method has been developed, and it is described in this report.

EXPERIMENTAL METHODS:

Advantage can be taken of difference in density between talc and amphiboles (see Table 1). The sample can be suspended in bromoform and centrifuged to float talc and settle denser minerals. However, the fine-grained nature of the ground ores and products brings colloidal forces into play which cause flocculation of the sample, and this renders a clean separation impossible.

Dispersion of talc in bromoform requires that the particles

be plated with organic molecules whose film thicknesses separate the grains sufficiently so that van der Waals forces cannot cause agglomeration. In addition, the plated grains must make up an oleophilic colloid, that is, they must be wettable by non-polar organic solvents (bromoform). Experiments were performed with various concentrations of butylamine hydrochloride, cetyltrimethylammonium bromide and benzethonium chloride monohydrate. The latter reagent was far superior in promoting dispersion in bromoform. The effectiveness of dispersion was judged by the time required for visual evidence of flocculation of the talc product.

The addition of benzethonium chloride monohydrate (hereafter, abbreviated BCM) lowered the density of the bromoform. Methylene iodide was added to bring the density back to desired levels. The proportions of sample and reagents used for the separations described in this report, are:

1 g talc,
2.0 g BCM,
20 ml Bromoform ($d=2.8$) and
8.4 ml Methylene iodide ($d=3.3$).

This mixture provides a density of 2.88 g/cm^3 , at 20°C , as measured by pycnometer. Attempts to raise the density by further additions of methylene iodide promoted flocculation. Consequently, future separations of amphiboles should be made at reagent concentrations similar to these.

Separations of amphiboles were made from ground talc product and from talc ore, provided by V. Zeitz of Windsor Minerals.

Talc was weighed into centrifuge tubes, and the reagents added in the proportions cited above. The tubes were shaken vigorously for approximately one minute, placed in a size 2 International Centrifuge, and centrifuged for 5 minutes at 500 RPM, followed by 5 minutes at 1800-2000 RPM. The centrifuge was allowed to slow with no braking (to minimize counter-rotating currents in the liquid) and the tubes were withdrawn and placed in racks for isolation of the heavy mineral fractions.

A glass rod was fitted with a rubber stopper, and this was carefully inserted into the tube and lowered to the bottom so as to isolate the heavy fraction (See Figure 1). The tube was decanted* and flushed with acetone from a polyethylene wash bottle. The plunger was removed, washed with acetone into the tube, and the tube was filled with acetone and shaken and centrifuged. The heavy fraction was washed twice more with acetone by means of the centrifuge, decanted, and dried overnight at 80°C. The tube with sample was cooled in a dessicator and weighed on an analytical balance. The concentrate at this stage consisted mostly of carbonate (magnesite plus some dolomite, see Plate 5). The carbonates were removed by acid dissolution as described below.

The sample tube was filled with 4NHCl and heated in a water bath for 2 hours at 80-90°C. The sample was centrifuged

* The reagent mixture cannot be saved. Slow decomposition occurs which liberates iodine and reduces the density. After 24 hours this process is sufficiently severe to lower the density below that of talc.

twice more to wash soluble salts out of the insoluble heavy mineral fraction. It was dried overnight at 80°C. The sample was scraped out of the tube, transferred to a vial, and the centrifuge tube was washed, dried, and weighed. From the three weights obtained, values were calculated for (1) percent heavy minerals, and (2) percent insoluble heavy minerals.

Talc ore and talc product, provided by V. Zeitz of Windsor Minerals, were run through this procedure. In addition, talc ore was spiked with known amounts of actinolite (ground and sized 2- ϕ by settling in water), and separated to test the efficiency of the method. The concentration of actinolite in the product concentrate was estimated by optical examination of the insoluble heavy mineral fraction. A simple estimate was made, and this was normalized to the total sample weight by means of the figure for the percentage of the total represented by the separate. Values for actinolite in the ore and in the spiked samples of ore were obtained by an X-ray fluorescence method that utilizes known amounts of potassium added as an internal standard. X-ray fluorescence methods were used because of the failure of various X-ray diffraction methods which were attempted. All of these had unacceptable precision ($\pm 100\%$), probably due in most part to the small (milligram) amounts of material available for analysis. Consequently, the X-ray fluorescence method provides the best means of measuring actinolite, although it would be useless for the determination of other fiberform amphiboles.

RESULTS:

Two-gram samples of ore and ore spiked with actinolite were separated and analyzed as described above. Results for these samples are summarized by Table 2. Table 3 shows concentrations of heavy fractions (mostly carbonate), and of actinolite in talc ore and talc product. Actinolite concentration in the product was measured for a 12 gram sample.

DISCUSSION:

The data of Table 2 show that (1) the total amount of heavy minerals separable from talc ore is reproduceable, and (2) the amounts of actinolite recovered from spiked samples agree quite closely with the actual amounts added. The agreement between actinolite found and actinolite nominal is about as good as can be expected, given the existence of errors of one or more milligrams that can arise as a result of the weighing procedures described above.

The samples studied contain actinolite as the dominant fiberform amphibole phase. This conclusion is based upon (1) the common occurrence of extinction angles of 15° , (2) the α refractive index of 1.616, and (3) the high calcium content of the residues which contained no other calcium-bearing minerals. However, some anthophyllite might be present in very small amounts (See Plates 7 and 8). It was identified on the basis of (1) parallel extinction, (2) extreme aspect ratio, and (3) lack of the length-parallel striations that characterize

actinolite.

The data of Table 1 show that cummingtonite and anthophyllite can have densities as low as 2.85, and these would float with talc in the liquids used (density = 2.88). But these low densities are for amphiboles that are pure magnesium end-members, and as such, are mineralogical curiosities that are very rare in nature. It is likely that the usual compositions of anthophyllite and cummingtonite would be closer in density to actinolite. Because all amphiboles have similar surface chemistry, the separation techniques described here would almost certainly work for their concentration from talc and talc ore matrices. In fact some progress has been made to that end, though the results are preliminary and can only be described as promising.

CONCLUSIONS:

1. Mixtures of bromoform, methylene iodide, and benzethonium chloride monohydrate provide a suitable heavy liquid for the centrifugal separation of fiberform amphiboles from talc in samples composed of clay to silt-sized grains.
2. The ore sample contains 2300 ppm actinolite, and the talc product contains ~170 ppm actinolite.
3. Actinolite is the dominant fiberform amphibole

in the ore and talc product provided by Windsor Minerals. Small amounts of anthophyllite may be present.

4. Calcium analyses on acid-treated heavy mineral residues serve as an effective means of determining actinolite.
5. The determination of crocidolite, cummingtonite, or anthophyllite in concentrates is probably best accomplished by a microscope method.

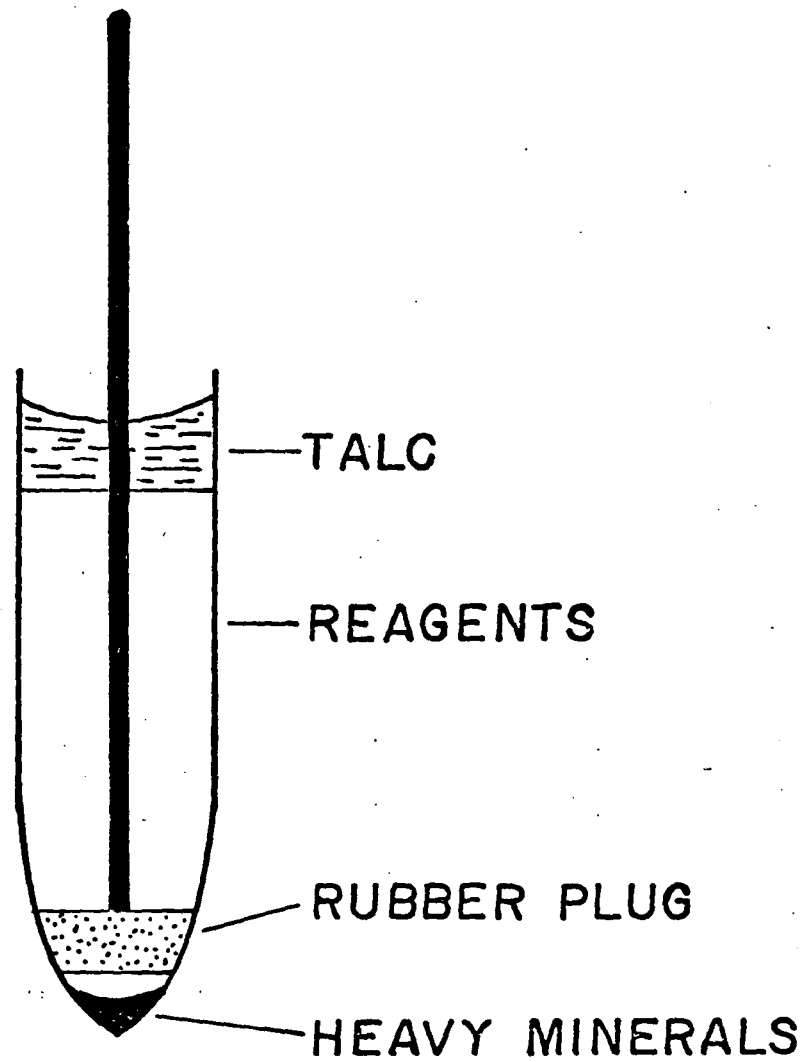


Figure 1. Apparatus for isolation of heavy minerals from centrifuge tube.

TABLE 1 - Densities of Common Fiberform Amphiboles
and Talc (Hurlbut, C.S., 1971, Dana's Manual of
Mineralogy, 18th Ed., Wiley).

Mineral	Density
Talc	2.7-2.8
Tremolite-Actinolite	3.0-3.3
Cummingtonite	2.85-3.2
Anthophyllite	2.85-3.2
Crocidolite	3.2-3.3

TABLE 2 - Residue Weights and Actinolite in Talc Ore and Actinolite-Spiked Talc Ore.

Sample	Heavy Minerals; mg/2g Sample	Acid Insoluble Heavy Minerals; mg/2g Sample	mg Actinolite found*	mg Actinolite nominal
Ore	478	14.3	4.6	---
Ore + 4.3 mg Actinolite	445	19.8	7.8	8.9
Ore + 8.6 mg Actinolite	458	20.4	9.8	13.2
Ore + 12.9 mg Actinolite	459	30.1	17.3	17.5

*Based on a nominal value of 9.7% Ca in actinolite,

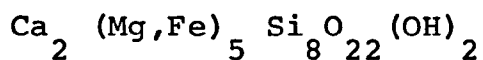


TABLE 3 - Heavy Fraction and Actinolite in Talc.

Sample	% Heavy Minerals (wCarbonate)	PPM Actinolite
Talc Ore	23.9	2300
Talc Product	--	170

APPENDIX

Photomicrographs have been taken of the minerals in talc and talc ores. Attempts have been made to catalog the dominant mineral species, and to depict the range in form characteristic of each mineral. For both talc product and talc ore, photos have been taken of (1) the bulk materials, (2) the heavy mineral fractions, and (3) the acid insoluble heavy mineral fractions. The photos below are accompanied by brief descriptions of each, and some mineral grains are appropriately labelled according to the following key.

Talc	T
Carbonate	C
Chromite	Cr
Actinolite	Ac
Anthophyllite	An
Epidote	Ep

Although the forms of the minerals shown on the photos are characteristic, many of the photos show non-representative mineralogical compositions. For example, a good deal of searching was required to find actinolite and carbonate grains in bulk talc products (Plates 14 and 11). Similarly, anthophyllite is extremely uncommon and considerable amounts of sample must be examined in order to find one grain (Plates 7 and 8).

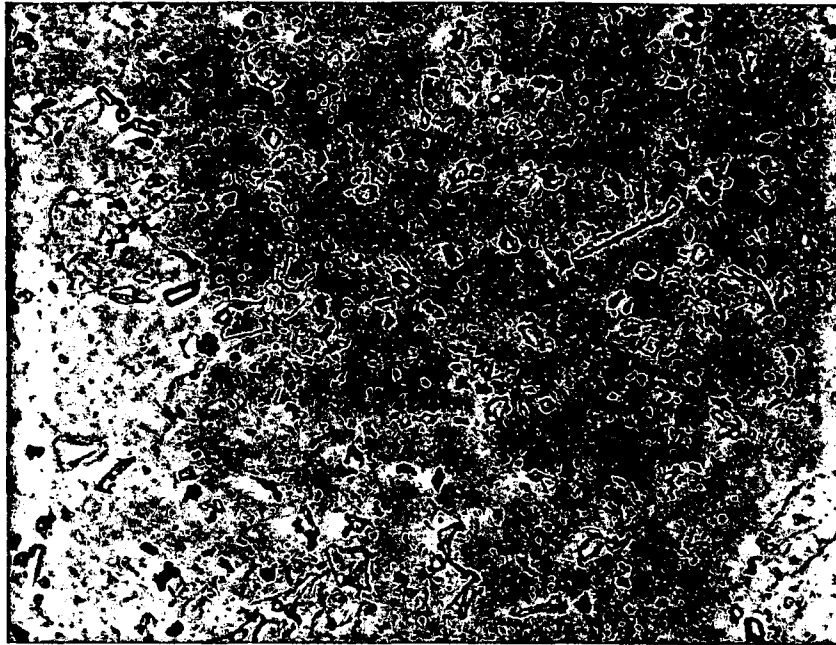


Plate 1

Talc Ore, Bulk; x 100; $n=1.503$
Low relief background is talc, high relief equant
grains are carbonate, and the long grain in the
upper right corner is actinolite.



Plate 2

Talc Ore, Bulk; x 400; $n = 1.503$
Platy talc, actinolite, and carbonate.
Note the length-striated character of actinolite;
this is characteristic.



Plate 3

Talc Ore, Bulk; x 400; $n=1.503$
Platy talc and carbonate. The talc
is inclusion-free and shows a well-
developed platy morphology.



Plate 4

Talc Ore, Heavy Fraction; x 400; $n = 1.503$
The field shows epidote, carbonate, talc,
chromite, and actinolite.

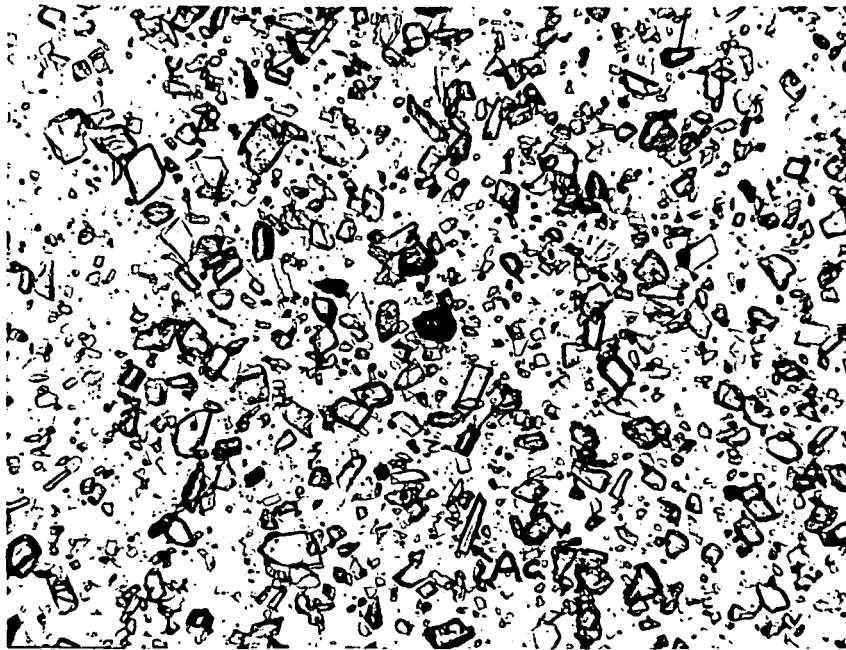


Plate 5

Talc Ore, Heavy Fraction; x 100; $n = 1.503$
This photo shows the general character of the typical heavy mineral fraction. Small amounts of talc are invisible in the background. Carbonate is the dominant mineral. Opaque grains are chromite, and actinolite is labelled Ac.



Plate 6

Talc Ore, Heavy Fraction; x 400; $n = 1.503$
This view shows mostly carbonate, some chromite
(opaque) and an actinolite grain of typical mor-
phology at the center.

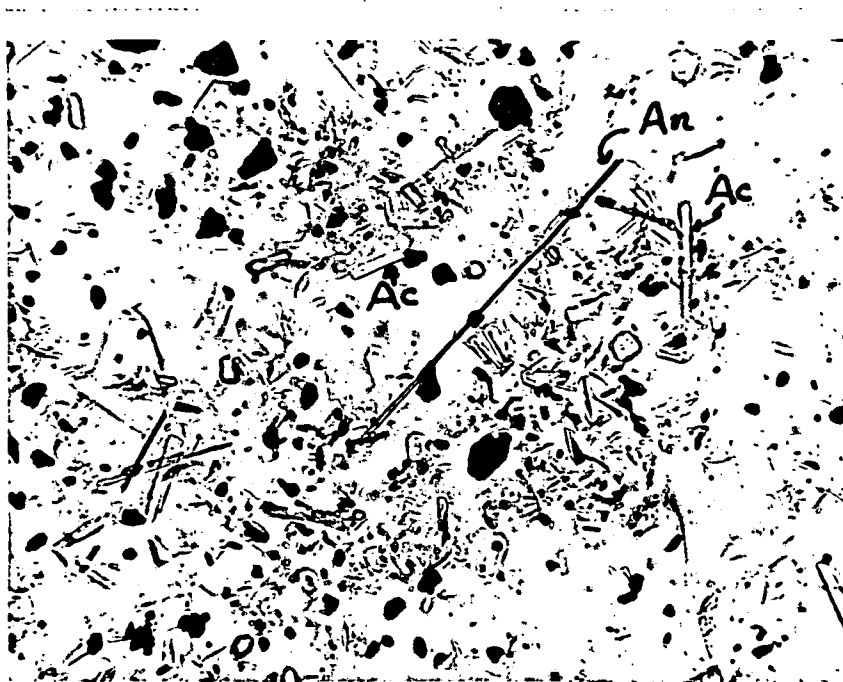


Plate 7

Talc Ore, Acid Insoluble Heavy Fraction; x 100; $n = 1.503$
Actinolite, talc, chromite, and a large anthophyllite fiber.
Note that much of the talc is of poor morphology and/or
is inclusion-filled.



Plate 8

Talc Ore, Acid Insoluble Heavy Fraction; x 400; $n = 1.503$
Inclusion-filled talc, actinolite and anthophyllite.



Plate 9

Talc Ore, Acid Insoluble Heavy Fraction; x 400; $n = 1.503$
Platy talc and actinolite. Note that the small equant
grains of actinolite could be easily mistaken for carbonate.

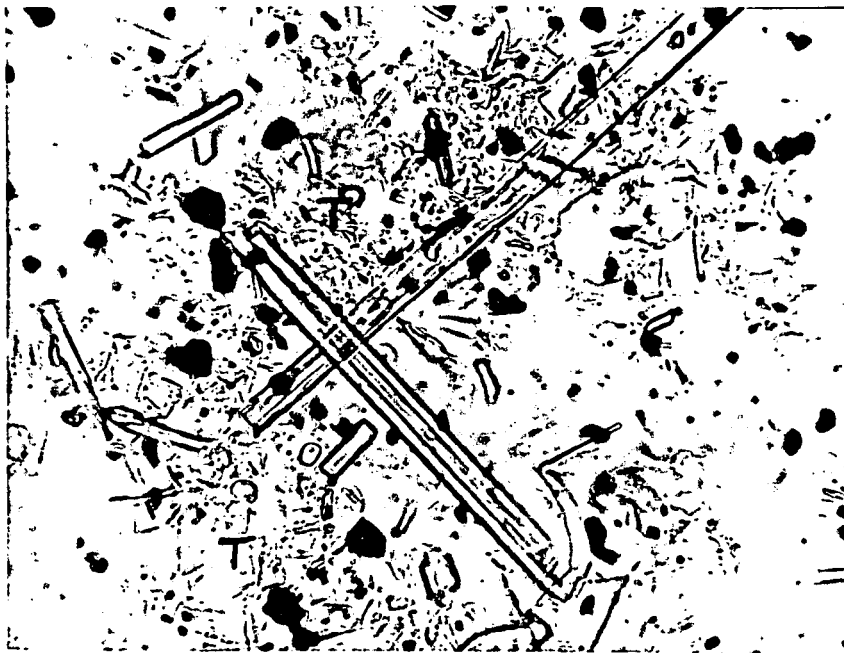


Plate 10

Talc Ore, Acid Insoluble Heavy Fraction; x 400; $n = 1.503$
Inclusion-filled talc with partial fibrous morphology,
platy talc, and characteristic grains of actinolite.



Plate 11

Talc Product, Bulk; x 100; n = 1.503
Platy talc with a few carbonate grains
(high relief).



Plate 12

Talc Product, Bulk; x 400; $n = 1.503$
Typical platy talc of good morphology.

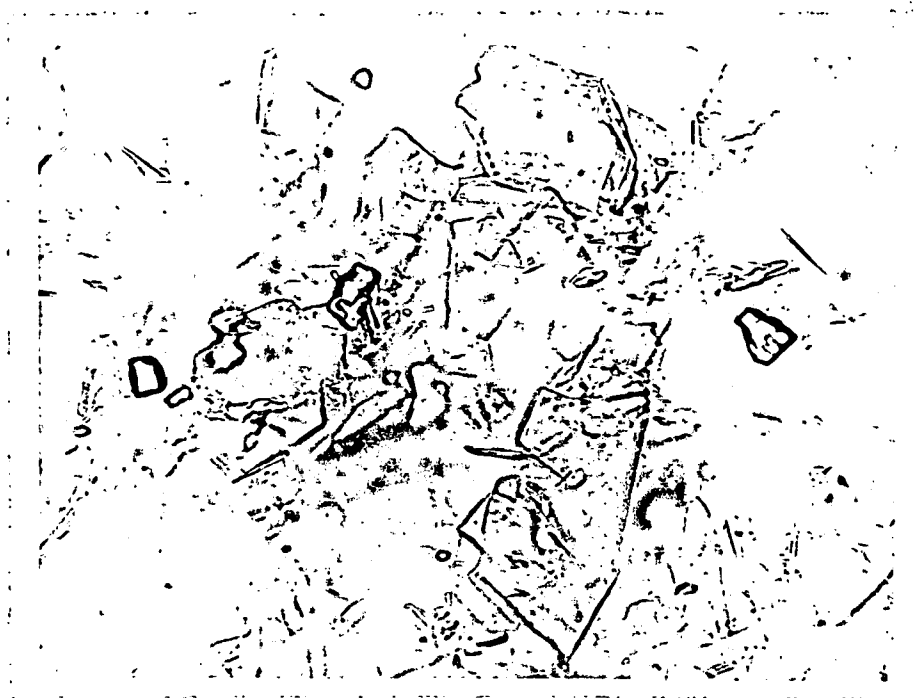


Plate 13

Talc Product, Bulk; $\times 400$; $n = 1.503$
Platy talc and carbonate.



Plate 14

Talc Product, Bulk; x 400; n = 1.503
Platy talc and one actinolite grain.



Plate 15

Talc Product, Heavy Fraction; x 400; $n = 1.503$
Typical field showing carbonate, talc, actinolite,
and chromite.

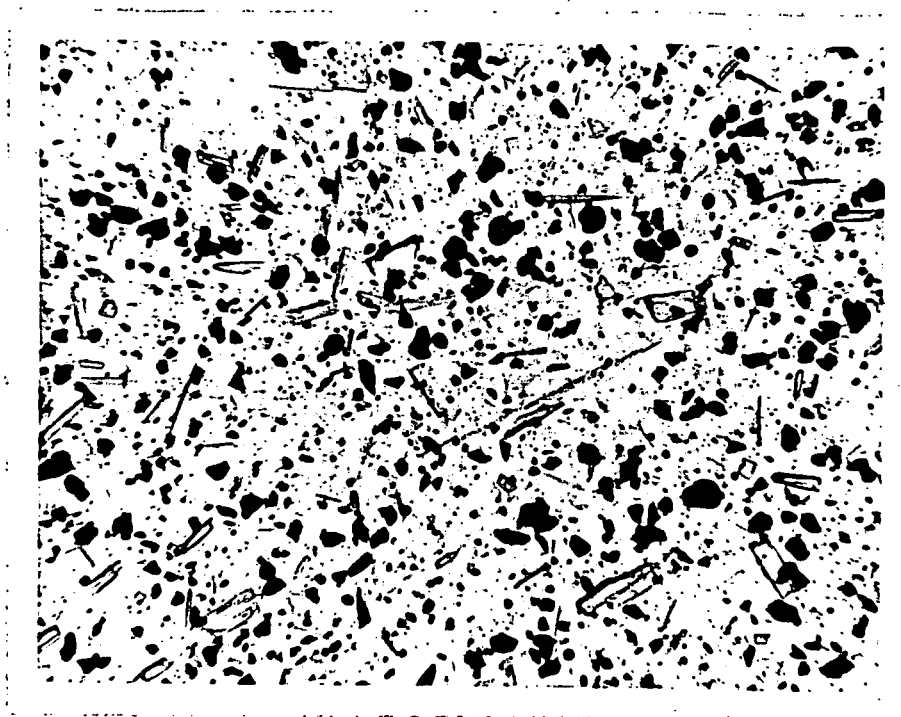


Plate 16

Talc Product, Acid Insoluble Heavy Fraction;
x 100; $n = 1.571$
Talc is invisible because of high index oil used.
Visible grains consist of chromite (opaque) and
actinolite. Note the large variation in aspect
ratio of the actinolite.

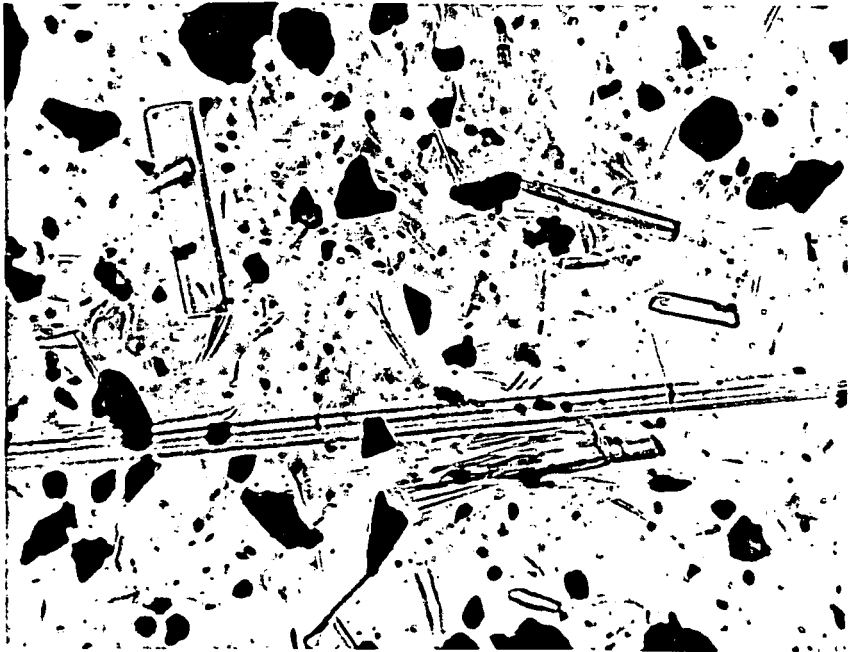


Plate 17

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.571$
Chromite and actinolite of varying morphology.
The characteristic striations are clearly visible
in the actinolite grains.



Plate 18

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.571$
Large actinolite grain with irregular shape.

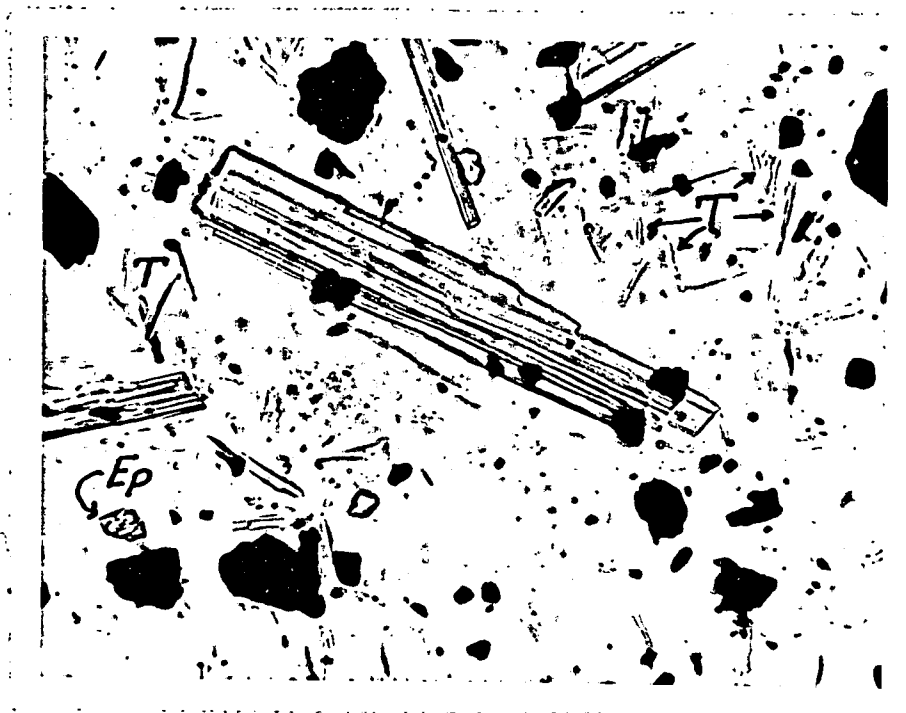


Plate 19

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.571$
Typical actinolite, fibrous talc, chromite and
epidote.



Plate 20

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.503$

Note the large compound grain (platy talc and
actinolite) at bottom center. Other minerals
are platy talc (very low relief) actinolite,
and chromite.

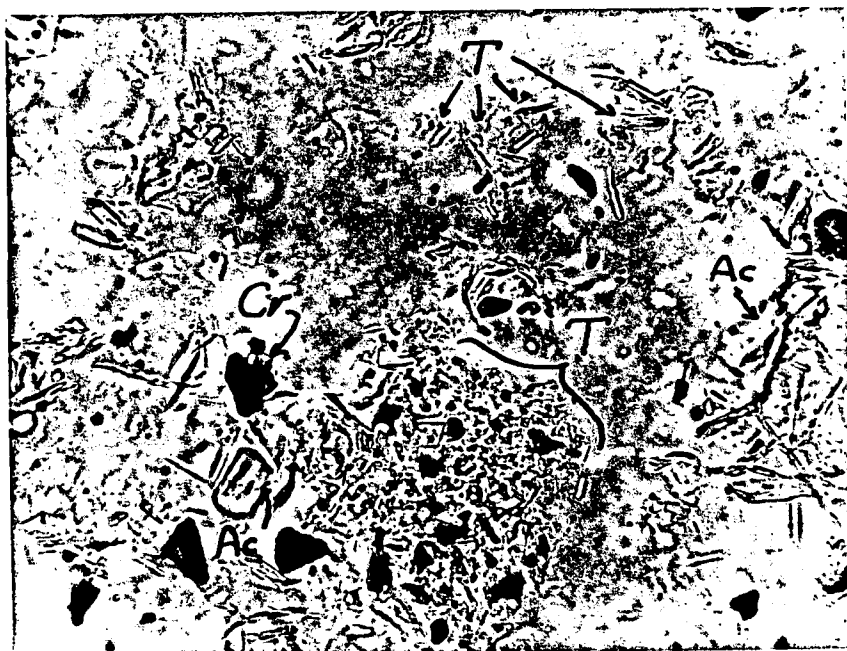


Plate 21

Talc Product, Acid Insoluble Heavy Fraction;
x 400; $n = 1.503$

Small talc fibers, platy talc, and inclusion-filled talc plus chromite and actinolite. It is the presence of grains such as the inclusion-filled talc that makes grain counting analysis difficult.